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Assessment of Emissions of Specific Compounds from a Resource Recovery Municipal Refuse Incinerator

Midwest Research Inst., Kansas City, MO

Prepared for

Environmental Protection Agency, Washington, DC

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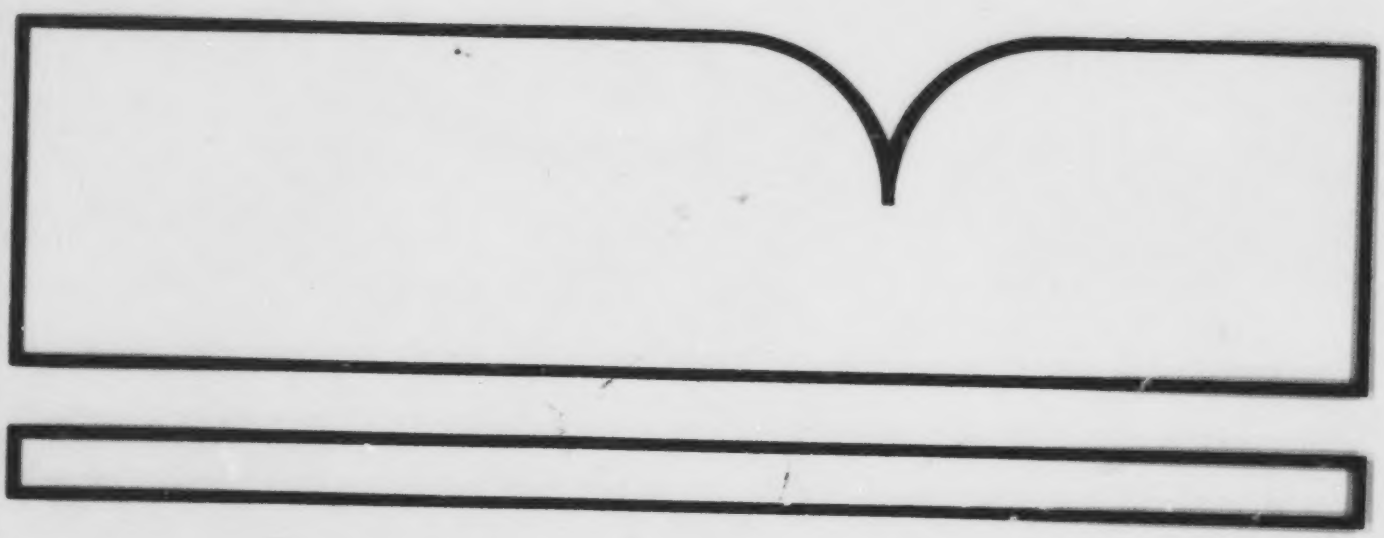

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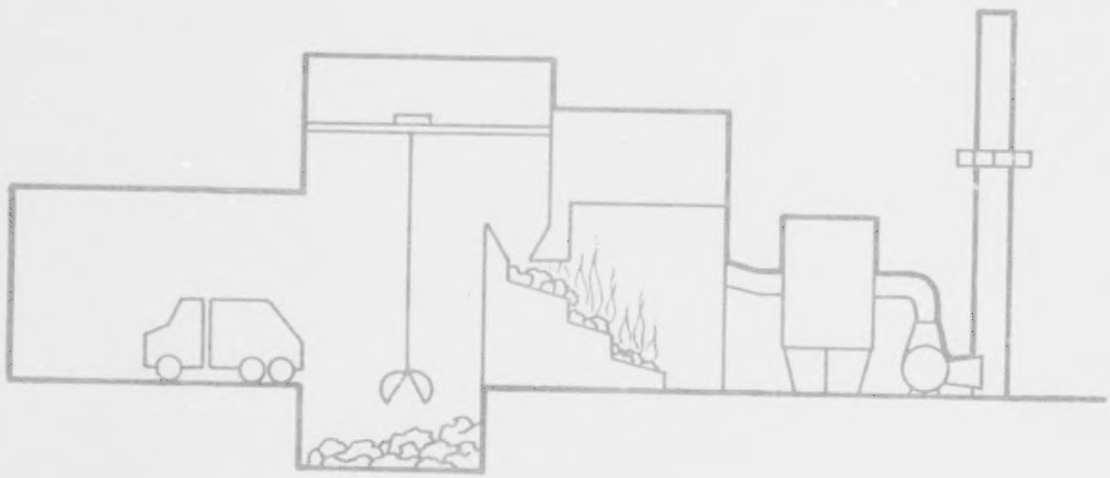

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This study was conducted as a part of a nationwide survey to determine organic emissions from major stationary combustion sources. The principal compounds of interest were polynuclear aromatic hydrocarbons (PAHs) and polychlorinated aromatic compounds, including polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins (PCDDs), and polychlorinated dibenzofurans (PCDFs). This report describes an assessment of emissions from a resource recovery municipal refuse incinerator.

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ASSESSMENT OF EMISSIONS OF SPECIFIC COMPOUNDS FROM A RESOURCE RECOVERY MUNICIPAL REFUSE INCINERATOR

by

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TASK 61
FINAL REPORT

EPA Contract No. 68-01-5915
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Prepared for

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DISCLAIMER

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PREFACE

This final report was prepared for the Environmental Protection Agency under EPA Contract No. 68-01-5915, Task 61. The task was directed by Dr. Clarence L. Haile. This report was prepared by Dr. Clarence L. Haile, Mrs. Ruth B. Blair, Dr. Robert M. Lucas (Research Triangle Institute), and Mr. Thomas Walker. Dr. John S. Stanley provided technical consultation and support for PCDD and PCDF analyses. Technical support was also provided by G. Scheil, R. Stultz, E. Olson, K. Hall, J. Pavelonis, D. Griffin, G. Radolovich, J. Onstot, and M. Wickham.

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LIST OF ABBREVIATIONS

acfm	-- Actual cubic feet per minute
dscf	-- Dry standard cubic feet
dscfm	-- Dry standard cubic feet per minute
dscm	-- Dry standard cubic meter
dscmm	-- Dry standard cubic meter per minute
EICP	-- Extracted ion current plots, constructed by computer from scanning gas chromatography/mass spectrometry data
ESP	-- Electrostatic precipitator
HRGC/MS	-- High resolution (fused silica capillary column) gas chromatography with low resolution mass spectrometry detection
HRGC/MS-SIM	-- HRGC/MS operating the spectrometer in a selected ion monitoring mode
HRGC/HRMS-SIM	-- HRGC/MS-SIM with the spectrometer operated at higher than unit resolution (e.g., 10,000 resolution)
PAHs	-- Polynuclear aromatic hydrocarbons
PCBs	-- Polychlorinated biphenyls
PCDDs	-- Polychlorinated dibenzo-p-dioxins
PCDFs	-- Polychlorinated dibenzofurans
PFK	-- Perfluorokerosene
PTFE	-- Polytetrafluoroethylene, e.g., Teflon®
THC	-- Total hydrocarbons

SECTION 1

INTRODUCTION

This study was conducted as a part of a nationwide survey to determine organic emissions from major stationary combustion sources. The principal compounds of interest were polynuclear aromatic hydrocarbons (PAHs) and polychlorinated aromatic compounds, including polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins (PCDDs), and polychlorinated dibenzofurans (PCDFs). This report describes an assessment of emissions from a resource recovery municipal refuse incinerator.

Previous tests conducted under this program include a pilot study of the variability of combustion source emissions and a nationwide survey of emissions from major coal-fired utility boiler plants. The pilot study involved 21 days of testing at a utility boiler plant co-fired with coal and municipal refuse-derived fuel and 11 days of testing at a resource recovery municipal refuse incinerator.[1] The variability of emissions (as determined from total organic chlorine in flue gas and ash samples) among test days and between the two plants was used to develop the sampling design for subsequent tests.[2] The emissions results from the pilot study and the nationwide survey of coal-fired utility boilers have been reported elsewhere.[1,3,4]

A summary of the results of the municipal refuse incinerator study is contained in Section 2 of this report. Section 3 presents recommendations for future work. A brief description of the incinerator is contained in Section 4. The sampling and analysis methods as applied to the plant are described in Sections 5 and 6. The field test data and analytical results are presented in Sections 7 and 8. Section 9 describes the analytical quality assurance results. The emissions results are summarized in Section 10.

SECTION 2

SUMMARY

This study was conducted as a part of a nationwide survey to determine organic emissions from major stationary combustion sources. The principal compounds of interest were polynuclear aromatic hydrocarbons (PAHs) and polychlorinated aromatic compounds, including polychlorinated biphenyls (PCBs), polychlorinated dibenzo-p-dioxins (PCDDs), and polychlorinated dibenzofurans (PCDFs). This report presents the results of emissions testing of a resource recovery municipal refuse incinerator.

All emissions (including flue gas, fly ash, and bottom ash) were sampled from a mass burn, refuse-fired steam generation plant. The furnace was designed to burn ∿ 125 tons/day and generated steam at 32,000 lb/hr. Daily flue gas samples (∿ 15 m^3) were collected from ports on the stack using modified EPA Method 5 trains. Plant background air was sampled during the flue gas testing and grab samples of ashes and quench waters were taken periodically according to a statistically derived 24-hr schedule. The total sample collection period was 5 days. The samples were extracted and analyzed using fused silica capillary gas chromatography with mass spectrometry detection (HRGC/MS).

PAHs and phthalates were identified in flue gas, fly ash, bottom ash, quench water, and background air samples. Naphthalene and acenaphthylene were the most abundant PAH compounds, averaging 620 and 220 μg/dscm, respectively, in flue gas samples. PCBs were identified in the flue gas and ash samples. Total PCBs in flue gas samples averaged 670 ng/dscm. Fly ash contained an average of 41 ng/g total PCBs. PCDD and PCDF compounds were identified in flue gas and fly ash. The mean concentrations of total PCDDs and PCDFs were 2,300 and 11,000 ng/dscm, respectively. Fly ash contained an average of 800 and 3,000 ng/g, respectively. The distribution of PCDD and PCDF homologs in the flue gas and fly ash samples were similar. Pentachloro homologs were most abundant.

Emission rates were determined for compounds identified in the flue gas samples by multiplying the concentrations by the flue gas flow rates. The emission rates calculated for PCBs, PCDDs, and PCDFs were 15, 51, and 250 mg/hr, respectively.

SECTION 3

RECOMMENDATIONS

1. Conduct emissions testing at additional refuse incineration facilities. Relatively few municipal refuse incineration units in the United States have been tested for emissions of specific hazardous organics. Additional data are needed to better characterize emissions from refuse incineration and allow assessment of the risks of current incineration practices.

2. Investigate relationships between emission rates for specific compounds and key plant design and operating parameters. The emissions and engineering data contained in this report, reported by other researchers, and resulting from testing at additional facilities should be compiled and evaluated, possibly using multivariant statistical analysis. The results of this analysis may provide relational information that could be used to design more efficient incineration units with lower emissions.

3. Determine the aqueous leaching potential of hazardous compounds identified in incinerator fly ash. Since incinerator fly ash is typically disposed by landfilling, information on the potential for leaching hazardous organics from ash with high concentrations of hazardous materials is needed to assess the potential for groundwater contamination.

4. Examine the computerized HRGC/MS data from this study for the presence of additional hazardous compounds. It is likely that the data archived for this study contains information on compounds (in addition to the target PAH, PCB, PCDD, and PCDF compounds) that would provide a more complete characterization of refuse incinerator emissions. Reanalysis of flue gas and fly ash extracts focusing on specific compounds could also expand the data available for examination. Target compounds should include nitropolynuclear aromatics, nitrogen heterocyclic aromatics, sulfur heterocyclic aromatics, and biphenylene.[5]

5. Develop a more complete inventory of PCDD and PCDF isomers. The variety of PCDD and PCDF isomers available for use as analytical standards is limited. The availability of additional standards, especially with chlorine in the 2, 3, 7, and 8 positions, would expand the number of isomers that could be uniquely determined.

SECTION 4

PLANT DESCRIPTION

The incineration plant consists of two identical units fired with raw (i.e., unprocessed) refuse. The incinerator sampled is shown diagrammatically in Figure 1. The refuse collection system supplying the unit services primarily residential urban and suburban neighborhoods. Large household appliances and other difficult to combust items are collected separately and disposed in a sanitary landfill.

The incinerator is charged with raw refuse from a storage pit by an overhead crane. The refuse burns without auxiliary fuel as it travels down a series of three inclined reciprocating grates. The residence time in the furnace is approximately 45 to 60 min. Unburned residue is discharged into a water-filled quench pit. Particulates removed from the flue gas with an electrostatic precipitator (ESP) are also conveyed into the quench pit. The pit is continuously dredged into a truck for landfill disposal.

The unit is designed to handle approximately 125 tons/day, producing steam at 32,000 lb/hr. The incineration process is somewhat susceptible to upsets caused by wet refuse, e.g., grass clippings and refuse collected during heavy rains. During stable operation, the firebox temperature is near 2300°F and the furnace wall temperature ranges from 1450 to 1550°F.

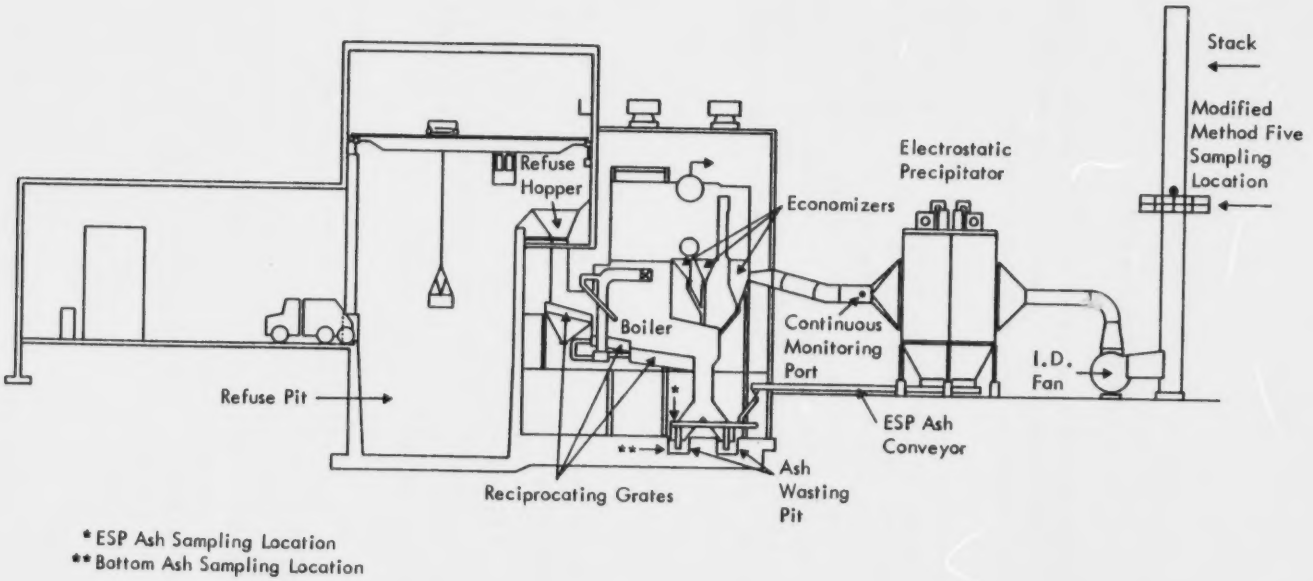

Figure 1. Incinerator cross-sectional view.

SECTION 5

SAMPLING METHODS

The general sampling methods used in this study are described in detail elsewhere.[5] This section presents an overview of the specific application of these methods to the resource recovery incinerator.

The general sample collection scheme is shown in Table 1. The sampling locations are indicated on the plant diagram in Figure 1. Samples were collected on 5 consecutive days. Flue gases and background air were sampled largely during daylight hours. Grab samples of ashes and quench water were collected according to a 24-hr schedule. Feed samples, i.e., raw refuse, were not collected due to the difficulty of obtaining representative specimens. The rigorous collection, homogenization and selection required to obtain representative specimens were beyond the scope of this study.

GASEOUS SAMPLES

Flue Gas

Flue gas samples were collected from two ports on the stack, located downstream from the ESP, using modified EPA Method 5 sampling trains. The modification consisted of a condensor to cool the gases and an adsorbent resin cartridge to retain organic vapors placed between the filter box and the first impinger. The cartridge was charged with 75 g of precleaned XAD-2 resin. Ice-chilled water was circulated through the condensor jacket and a jacket around the cartridge during sample collection.

A single flue gas sample was collected on each sampling day using two trains. The trains were operated at isokinetic sampling rates and were traversed (as specified in EPA Method 5) until roughly 5 to 7.5 m^3 were collected in each train. The locations of the sampling points are presented in Table 2 and are shown on a diagram of the stack cross section in Figure 2. Each daily sample consisted of the particulate catches, resin cartridges, and rinses (of probe and train components forward of the first impinger) from both trains. The contents of the first impinger in each train were also recovered to check for analyte breakthrough.

Plant Background Air

A single plant background air sample was collected each sampling day using a resin cartridge (identical to that used in the flue gas trains), a pump, and a dry gas meter. The sampling system was located near the air inlet to the furnace, on a walkway near the overhead crane over the refuse pit.

TABLE 1. SAMPLES COLLECTED, SAMPLING LOCATIONS, AND COLLECTION FREQUENCIES

Sample type	Location	Collection frequency
Gaseous samples		
Flue gas	Ports on stack	1/day
Plant background air	Catwalk above refuse pit	1/day
Solid samples		
Bottom ash	Conveyor from sluice trench	6/day
Fly ash	Conveyor to sluice trench	6/day
Aqueous samples		
Quench water effluent	Overflow weirs from sluice trench	6/day
Quench water influent	Recycled water holding tank	2/day

TABLE 2. MODIFIED METHOD 5 TRAIN SAMPLE POINT LOCATIONS

Traverse point no.	Fraction of stack I.D. (%)	Distance from stack wall (in.)
1	4.4	2.13
2	14.6	7.00
3	29.6	14.25
4	70.4	33.75
5	85.4	41.00
6	95.6	45.88

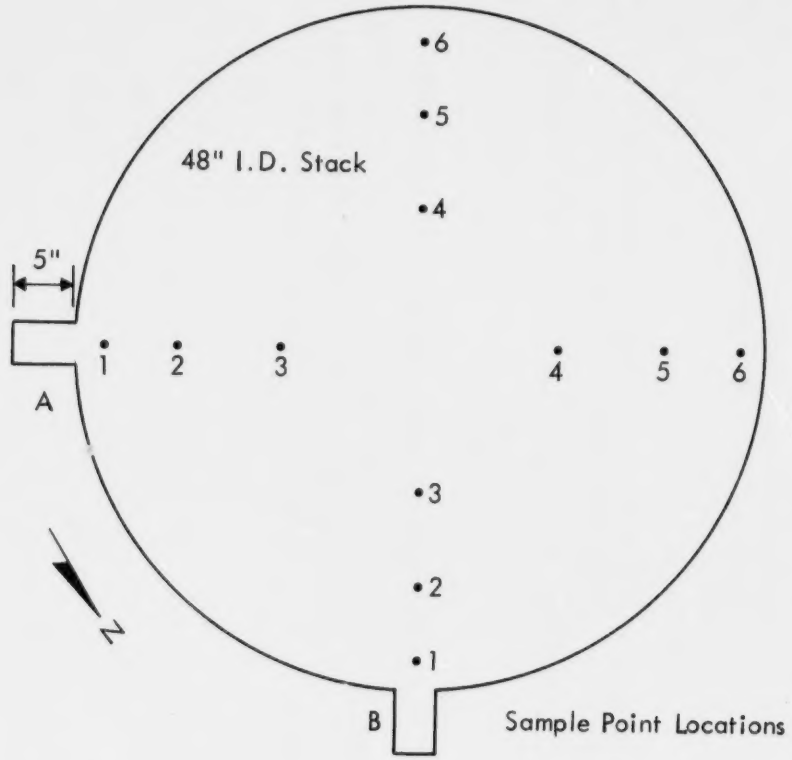

Figure 2. Flue gas sampling point locations - stack cross section.

The sampling rate was maintained at 0.70 to 0.75 ft^3/min (0.020 to 0.021 m^3/min) until 6.5 to 7.5 m^3 were collected.

Continuous Monitoring

Ports for continuous monitoring of the combustion gas composition were located immediately upstream from the ESPs (Figure 1).

SOLID AND AQUEOUS SAMPLES

Collection Schedule

The schedule used for collection of the grab samples, i.e., fly ash, bottom ash, quench water influent, and quench water effluent, was constructed based on the variability of organic emissions observed for a two-plant study.[1] The statistical analysis of emissions variability has been described elsewhere.[2]

The objectives of the grab sample collection schedule were:

(1) simplicity of implementation in the field, and

(2) acceptable statistical (probability sampling) methods.

To accomplish these objectives, the sampling protocol must involve acquiring specimens from the different media at random times (because levels of target compounds may vary over time), but have sufficient structure to assure practical application in the field. For example, because of limited field personnel, it is impractical to collect specimens from different media simultaneously (or within a very short time period). The protocols described below satisfied the objectives.

Structure for the sampling protocols was developed by first defining an ordering to the media. The ordering was bottom ash (BA), fly ash (FA), quench water effluent and water influent. A one-half hour time period between specimen acquisition by media was established to assure adequate time for field personnel to properly conduct the specimen acquisition, labelling, and storage.

Proper randomization over time was accomplished in a method that was compatable with anticipated specimen compositing schemes and also included structure to maintance simplicity in application in the field. The sampling period of five days was first partitioned into ten nonoverlapping periods referred to as strata. The ten strata are defined in Table 3. To select random times, four-hour intervals were assigned 16 time points, 15 minutes apart. For example, 0000, 0015, 0030, ..., 0345 (military time). Fifteen (15) minute intervals were judged to provide adequate time resolution because errors resulting from variation in the true levels of the target compounds would be small compared to the anticipated analytical precision.

TABLE 3. TIME STRATA FOR SPECIMEN COLLECTION

Stratum	Day	Military time[a]
1	1	0000-4000, 8000-1200, 1600-2000
2	1	4000-8000, 1200-1600, 2000-2400
3	2	0000-4000, 8000-1200, 1600-2000
4	2	4000-8000, 1200-1600, 2000-2400
5	3	0000-4000, 8000-1200, 1600-2000
6	3	4000-8000, 1200-1600, 2000-2400
7	4	0000-4000, 8000-1200, 1600-2000
8	4	4000-8000, 1200-1600, 2000-2400
9	5	0000-4000, 8000-1200, 1600-2000
10	5	4000-8000, 1200-1600, 2000-2400

a Time intervals include the left end point and exclude the right end point.

One of the 16 time points was selected at random for each stratum yielding 10 random points. The specimen collection times were assigned using the ten random points, the ordering of the media, and the 30 minute interval criterion. The general formulas for determining the specimen collection times are given in Table 4. Because each stratum consisted of three 4-hour periods, 400, 800, 1200, 1600, or 2000 (military time) had to be added to the general formula as appropriate. Table 5 presents the detailed specimen collection schedule by media. Because the quench water influent was anticipated to be reasonably homogeneous, one sample per stratum was judged to be adequate. One time period per stratum was selected randomly for collection of quench water influent.

TABLE 4. GENERAL FORMULAS FOR DETERMINING SPECIMEN COLLECTION TIMES

Media	Formula[a]
Bottom ash	r_i
Fly ash	$r_i + 30$
Quench water effluent	$r_i + 100$
Quench water influent	$r_i + 130$

a Randomly selected time for stratum i expressed as military time.

TABLE 5. SAMPLING SCHEDULE BY MEDIA (MILITARY TIME)

Day	Stratum	Bottom ash	Fly ash	Quench water effluent	Quench water influent[a]
1	1	0215	0245	0315	0345
	2	0730	0400	0430	
	1	1015	1045	1115	
	2	1530	1200	1230	1300
	1	1815	1845	1915	
	2	2330	2000	2030	
2	3	0145	0215	0245	0315
	4	0715	0745	0415	
	3	0945	1015	1045	
	4	1515	1545	1215	1245
	3	1745	1815	1845	
	4	2315	2345	2015	
3	5	0000	0030	0100	0130
	6	0645	0715	0745	
	5	0800	0830	0800	
	6	1445	1515	1545	1215
	5	1600	1630	1700	
	6	2245	2315	2345	
4	7	0230	0300	0330	0000
	8	0730	0400	0430	
	7	1030	1100	1130	
	8	1530	1200	1230	1300
	7	1830	1900	1930	
	8	2330	2000	2030	
5	9	0330	0000	0030	0100
	10	0730	0400	0430	
	9	1130	0800	0830	
	10	1530	1200	1230	1300
	9	1930	1600	1630	
	10	2330	2000	2030	

a One sampling period within each stratum was selected randomly (equal probabilities). Because the water influent was anticipated to be reasonable homogeneous, one specimen per stratum was judged to be adequate.

ESP Ash

Fly ash samples were collected six times each sampling day from the conveyor line immediately above the quench pit.

Bottom Ash

Bottom ash samples were collected six times each sampling day from the conveyor chain used to transfer drained quench pit residue into trucks for disposal. Relatively large items (> 4 cm) were rejected from the samples taken. Since ESP and economizer hopper ash was also wasted via the quench pit, bottom ash samples contained both furnace residue and fly ash.

Quench Water Effluent

The effluent from the quench pit was sampled six times each sampling day from an overflow weir at the quench pit. The quench water recycle system is shown diagrammatically in Figure 3.

Quench Water Influent

The influent to the quench pit was sampled twice each sampling day. Samples were bailed from an access port between the water treatment (recycle) system and the quench pit.

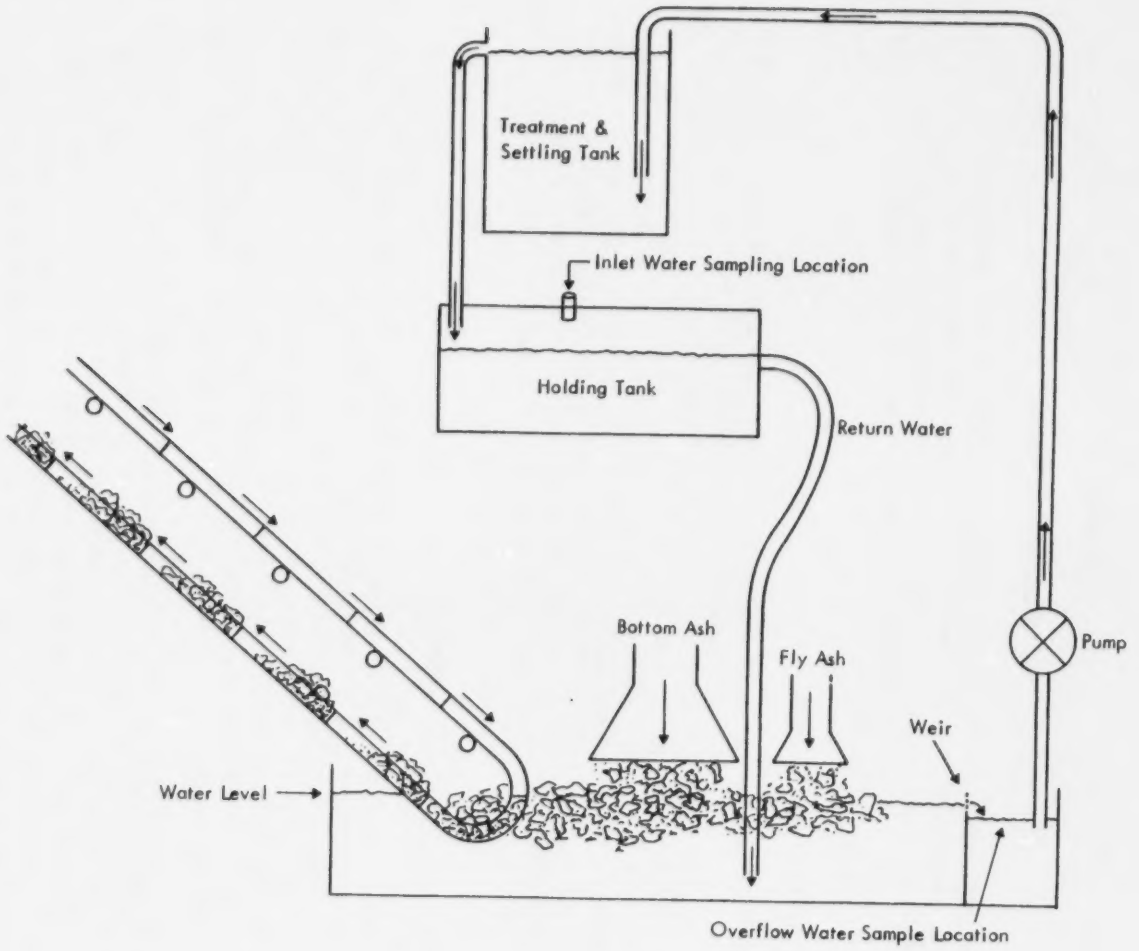

Figure 3. Quench pit and water recycle system.

SECTION 6

ANALYSIS METHODS

The general procedures for sample preparation and analysis are described in detail elsewhere.[5] This section provides descriptions of specific procedures used for sample compositing, extract compositing, and extract cleanup as well as other details related to the analyses of the samples from the resource recovery incinerator.

GENERAL ANALYTICAL SCHEME

Sample preparation and analysis followed the general analytical scheme presented in Figure 4. The samples were spiked with surrogate compounds just prior to extraction. Extracts were analyzed by fused silica capillary gas chromatography/mass spectrometry (HRGC/MS) to provide information on the recovery of PAH surrogate compounds and quantitation of polynuclear aromatic hydrocarbons, phthalates, and other major components of the sample extracts. Extracts were analyzed for PCBs, PCDDs, and PCDFs by HRGC/MS-SIM (selective ion monitoring). HRGC with high resolution mass spectrometry (HRGC/HRMS-SIM) was used to confirm the identification and to quantitate tetrachlorodibenzo-p-dioxins in selected extracts.

SAMPLE COMPOSITING AND EXTRACTION

Ash and aqueous effluent samples were composited prior to analysis to form two individual 5-day plant composites for each sample type. The statistical design of the sampling schedule defines the appropriate statistical analysis of the data. In order to assure that adequate information is retained to properly estimate the level of input and emissions of the target compounds and their corresponding variances, the specimen composition protocol must be nested within the statistical design structure, namely, the strata.

Specimens collected at different time periods within each stratum can be composited into one aliquot for chemical analysis. This will yield an estimate of the level of the target compound for the stratum that have a one-to-one correspondence with the randomly selected time points. Hence, equal weights of samples from collection times 1, 3, and 5 were combined to form daily composite A for each day, while 2, 4, and 6 were combined to form daily composite B. These were further composited by combining equal weights of the daily composites to form plant composites A and B. Equal volumes of the first influent water sample each day formed plant composite A. Plant composite B for influent water was similarly derived.

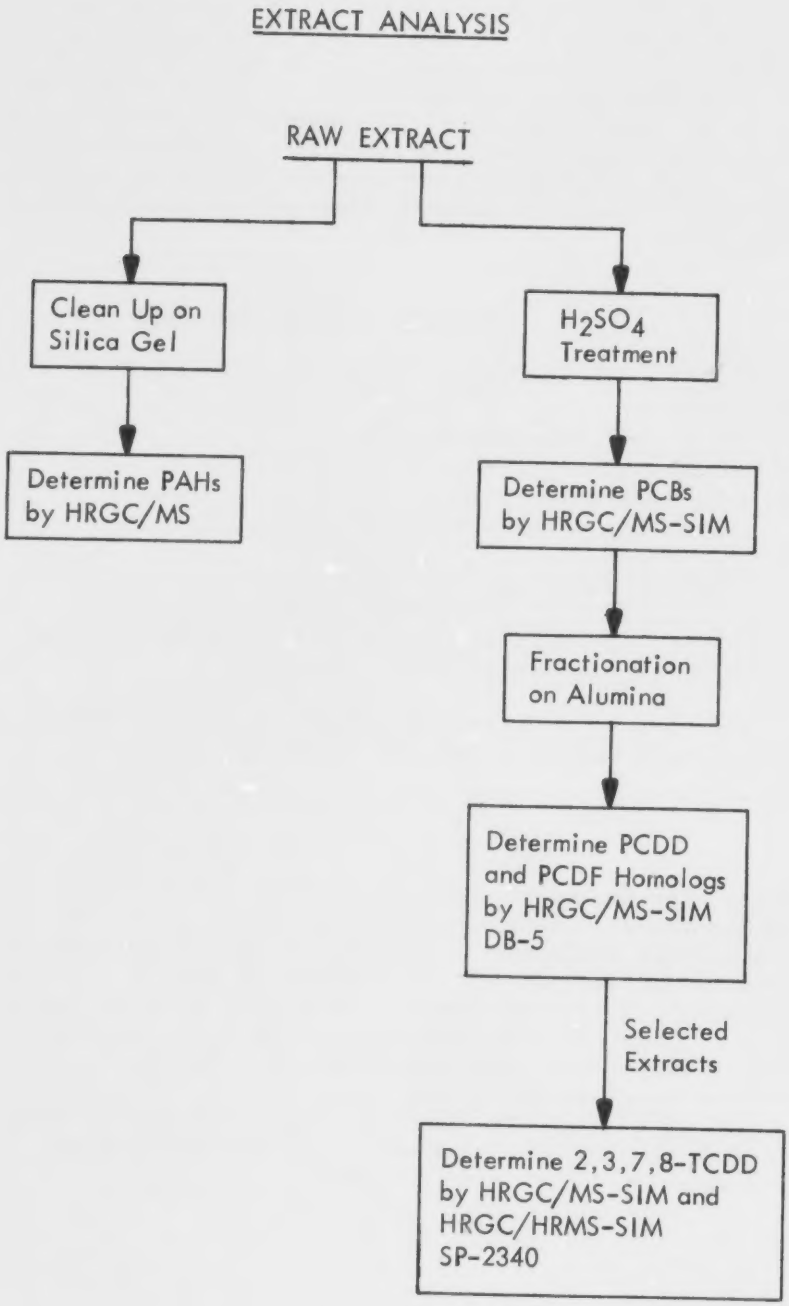

Figure 4. General analytical scheme.

Immediately prior to extraction, all composite and other grab samples prepared for extraction were spiked with the compounds listed in Table 6. The surrogate spiking compounds were selected from commercially available stable labeled compounds to represent specific classes of the target analytes. Naphthalene-d_8 and chrysene-d_{12} were selected to represent small and large PAH compounds. Naphthalene is the most volatile of the target analytes. Hence, naphthalene-d_8 recoveries may provide an indication of maximum losses attributable to volatilization during extraction and extract concentration. Chrysene-d_{12} is the least volatile of the surrogate compounds. General chlorinated aromatics were represented by 1,2,4,5-tetrachlorobenzene-$^{13}C_6$. Pentachlorophenol-$^{13}C_6$ was selected to represent the most polar of chlorinated phenols. The four labeled PCB and PCDD compounds were selected to represent those compound classes.

TABLE 6. SURROGATE SPIKING COMPOUNDS

50 µg	pentachlorophenol-$^{13}C_6$
50 µg	chrysene-d_{12}
50 µg	naphthalene-d_8
50 µg	tetrachlorobenzene-$^{13}C_6$
100 ng	4-chlorobiphenyl-$^{13}C_6$
250 ng	3,3',4,4'-tetrachlorobiphenyl-$^{13}C_{12}$
400 ng	2,2',3,3',5,5',6,6'-octachlorobiphenyl-$^{13}C_{12}$
500 ng	decachlorobiphenyl-$^{13}C_{12}$
100 ng	2,3,7,8-tetrachlorodibenzo-p-dioxin-$^{37}Cl_4$
100 ng	octachlorodibenzo-p-dioxin-$^{13}C_{12}$

Each daily flue gas sample consisted of the cyclone catch and filter (combined in the field), adsorbent resin, and probe rinse from two modified Method 5 trains. The surrogate compounds were spiked into one of the resins for each sampling day and into either a filter on days 1, 3 and 5, or a probe rinse for each day. Following separate extraction of each component, the probe rinse and filter extracts from both trains were combined. The resin extracts from the two trains were also combined. Hence, the flue gas extracts consisted of a probe rinse and filter catch extract and a resin extract for each of the five test days. The first impinger contents for each of the sampling trains from days 2 and 4 were extracted and analyzed separately to test for breakthrough of analytes from the resin.

All solid samples, i.e., resin, particulate catch, bottom ash, and ESP ash, were Soxhlet extracted with benzene. All aqueous samples, i.e., probe rinse, impinger and quench waters, were batch extracted with three portions of cyclohexane. All extracts were dried by passage through a short column of precleaned anhydrous sodium sulfate and concentrated to ∿ 5 mL using Kuderna-Danish evaporators. The extracts were further concentrated to 1.0 mL under a gentle stream of dry nitrogen. The concentrated extracts were then split into two equal aliquots for subsequent analysis.

EXTRACT CLEANUP

Silica Gel Chromatography

One aliquot of each extract (except aqueous and first impinger sample extracts) were cleaned by adsorption column chromatography prior to scanning HRGC/MS analysis. This cleanup procedure was adapted from methods developed by MRI for cleanup of sludge extracts.[6] Twenty-gram aliquots of freshly prepared silica gel (70 to 230 mesh, Soxhlet extracted with dichloromethane, dried at 110°C and deactivated with 1% water) were slurried with hexane and transferred to 14.5 x 250 mm chromatography columns. Individual extracts were added to 2-g aliquots of silica gel and evaporated to dryness. The extracts were then placed at the top of the columns and eluted according to the following scheme.

Fraction 1 = 20 mL hexane
Fraction 2 = 80 mL hexane
Fraction 3 = 50 mL 10% benzene in hexane
Fraction 4 = 50 mL 50% benzene in hexane
Fraction 5 = 150 mL 10% acetone in benzene
Fraction 6 = 40 mL methanol

Fraction 1 from each column was discarded. Fractions 2 to 5 were composited prior to scanning HRGC/MS analysis. Fraction 6 was collected separately and held to check for late elution of certain compounds. Table 7 shows the recoveries observed for the target PAH and phthalate compounds spiked onto silica gel and eluted according to the above scheme.

Acid Treatment

The remaining aliquot of each sample extract was cleaned by acid treatment prior to HRGC/MS-SIM analysis. Each extract aliquot was diluted to 5 mL with benzene and washed with 5 mL of concentrated sulfuric acid (preextracted with benzene) for approximately 1 min. The phases were allowed to separate and the organic layer was removed. The H_2SO_4 layer was extracted with three 2-mL aliquots of fresh benzene. The benzene was removed and combined with the original benzene fraction. If the H_2SO_4 layer was highly colored, the treatment was repeated. Finally, the benzene was back-extracted with several drops of distilled water to remove any residual acid. The cleaned extracts were then dried and concentrated. The recoveries for PCB surrogates spiked into benzene and acid-extracted twice are shown in Table 8.

TABLE 7. RECOVERIES FOR COMPOUNDS CHROMATOGRAPHED ON SILICA GEL BY THE PROCEDURE USED TO CLEAN SAMPLE EXTRACTS

Compound	Mean[a,b] % recovery	Standard deviation
Naphthalene	87	43
Acenaphthylene	79	28
Dimethyl phthalate	85	33
Acenaphthene	102	41
Fluorene	85	34
Diethyl phthalate	92	19
Phenanthrene	61	18
Di-n-butyl phthalate	90	19
Fuoranthene	90	20
Pyrene	86	17
Butylbenzyl phthalate	95	15
Chrysene	94	15
Bis[2-ethylhexyl]phthalate	89	18
Di-n-octyl phthalate	92	13
Benzo[g,h,i]perylene	97	26

a Taken from three individual batches of silica gel.

b Spike level was 25 µg.

TABLE 8. RECOVERY OF PCB SURROGATE COMPOUNDS FROM SULFURIC ACID TREATED EXTRACTS

Compound	Recovery (%)
Chlorobiphenyl-$^{13}C_6$	99
3,3',4,4'-Tetrachlorobiphenyl-$^{13}C_{12}$	100
2,2',3,3',5,5',6,6'-Octachlorobiphenyl-$^{13}C_{12}$	78
Decachlorobiphenyl-$^{13}C_{12}$	81

Alumina Chromatography

Following PCB analyses on the acid-treated extract aliquots, the aliquots were fractionated on microalumina columns to remove PCBs. The procedure has been described in detail elsewhere.[7] Briefly, columns were prepared by placing 1 g of aluminum oxide (Woelm Pharma, Eschwege, West Germany), W 200 basic, activity grade super 1, in a disposable Pasteur pipet with a small plug of glass wool. The extract (at 0.5 mL) was added to the top of the column. Each column was eluted with 10 mL of 2% dichloromethane in hexane, and the eluent was discarded. The column was then eluted with 10 mL of 50% dichloromethane in hexane. The eluent was collected and concentrated to 1.0 mL for analysis. Spiked blank columns were run each day along with samples.

EXTRACT ANALYSIS

Scanning HRGC/MS

The sample extracts were analyzed by scanning HRGC/MS to identify and quantify PAHs, phthalates, and any chlorinated compounds that might be present. Table 9 lists the target PAH and phthalate compounds. The gas chromatography and mass spectrometer instrumental parameters for the scanning HRGC/MS analyses are given in Table 10. Anthracene-d_{10} (20 ng/μL) was added to sample extracts and standards prior to scanning HRGC/MS to serve as internal standard for quantitation. The surrogate compound standard (50 ng/μL for PAHs) and a 25 ng/μL PAH-phthalate standard were analyzed at least once per day with the sample extracts.

The PAHs, phthalates, and surrogate compounds were identified using three extracted ion current plots (EICPs) for each specific compound. The criteria for compound identification were coincident peaks in all EICPs at the appropriate retention time with the characteristic response ratios. Compounds identified were quantified by comparing the EICP response for the most abundant ion with the most abundant ion of the internal standard (anthracene-d_{10}) and using the response factor for these two ions determined from the standard solutions.

HRGC/MS-SIM for PCBs

Extracts of grab samples, plant background air, and flue gas were analyzed for PCBs using a specialized HRGC/MS-SIM procedure, selected mass range scan HRGC/MS. That is, the mass spectrometer was scanned over the m/e range of the molecular cluster for each of the chlorobiphenyls. The specific operating parameters are listed in Table 11. In order to improve sensitivity, scan ranges were switched according to a preset program during the course of the HRGC/MS run so that only two sets of chlorobiphenyl ions were monitored simultaneously. The specific time points for switching the ion sets were selected based on the elution times for chlorobiphenyl compounds in a mixture of Aroclor® 1248, 1254, and 1260. Ions for monochloro- and dichlorobiphenyl were monitored from the initiation of the run until a time after the elution of monochlorobiphenyl but before the elution of trichlorobiphenyl. At that time, the ion set was switched to monitor for dichloro- and trichlorobiphenyl.

TABLE 9. TARGET PAH ANT PHTHALATE COMPOUNDS

PAHs	Phthalate esters
Naphthalene	Dimethylphthalate
Acenaphthylene	Diethylphthalate
Acenaphthene	Di-n-butylphthalate
Fluorene	Butylbenzylphthalate
Phenanthrene	Bis(2-ethylhexyl)phthalate
Anthracene	Di-n-octylphthalate
Fluoranthene	
Pyrene	
Chrysene	
Benzo[k]fluoranthene	
Benzo[a]pyrene	
Dibenz[a,h]anthracene	
Benzo[g,h,i]perylene	

TABLE 10. INSTRUMENT AND OPERATING PARAMETERS FOR SCANNING HRGC/MS ANALYSIS

Instrument	Finnigan MAT 311-A/Incos
Column	15-m fused silica, wall-coated with SE-54 or DB-5
Column temperature	80°C for 2 min, then to 325°C at 10°C/min
Carrier gas	Helium at 2.5 psi
Injector	J & W on-column (1 μL injection)
Scan range	m/e 32-425
Scan rate	1.5 sec/scan
Mass resolution	1,000 (m/Δm, 10% valley)

TABLE 11. INSTRUMENTAL PARAMETERS AND MASS RANGES USED FOR HRGC/MS-SIM ANALYSES OF PCBs

Instrument	Finnigan 4023
Column	15 m fused silica, wall-coated with DB-5
Column temperature	80°C for 2 min, then to 325°C at 8°C/min
Carrier gas	Helium at 2.5 psi
Injector	J&W on-column (1 μL injection)
Scan rate	1 sec/scan
Mass resolution	unit

Scan ranges

No. chlorines	Mass range scan (amu)	Retention time monitored (min)a
1	187.5 - 188.5	13.0 - 14.5
2	221.5 - 226.5	13.0 - 14.5
3	255.5 - 262.5	13.0 - 16.9
4	289.5 - 298.5	14.5 - 18.2
5	323.5 - 334.5	16.9 - 20.0
6	357.5 - 366.5	18.2 - 22.2
7	391.5 - 400.5	20.0 - 23.1
8	425.5 - 434.5	22.2 - 25.0
9	459.5 - 468.5	23.1 - 26.6
10	493.5 - 502.5	25.0 - 26.6

a Determined by analyzing a mixed Aroclor standard and scanning HRGC/MS.

This sequence was continued throughout each run. Hence, the last set of ions monitored was for nonachlorobiphenyl and decachlorobiphenyl. Positive responses to any of the PCB isomers in the composite extracts were confirmed when the peaks for the ion plots for two ions were coincident with responses in the proper ratios. PCB isomers identified were quantitated using area response factors for specific isomers with the same chlorine number. Standard solutions containing the isomers listed in Table 12 were analyzed at the following concentration ranges: 25-125, 50-250, and 100-500 pg/μL.

TABLE 12. PCB COMPOUNDS USED FOR QUANTITATION STANDARDS

4,4'-Dichlorobiphenyl
2,3,5'-Trichlorobiphenyl
2,4,2',4'-Tetrachlorobiphenyl
2,3,4,5,6-Pentachlorobiphenyl
2,3,4,2',3',4'-Hexachlorobiphenyl
2,3,4,5,6,2',5'-Heptachlorobiphenyl
2,3,4,5,2',3',4',5'-Octachlorobiphenyl
Decachlorobiphenyl

HRGC/MS-SIM for PCDDs and PCDFs

Sample extracts were also analyzed by HRGC/MS-SIM for PCDDs and PCDFs. The instrument and operating parameters are listed in Table 13. Perfluorokerosene (PFK) was used to obtain stable mass assignments during PCDD and PCDF analyses. Analyses for the entire range of PCDDs and PCDFs required four injections of each extract. Mono- through tri- PCDDs and PCDFs were determined in the first run. Three subsequent runs were used to determine tetrachloro compounds, penta- and hexachloro compounds, and hepta- and octachloro compounds, respectively.

HRGC/HRMS-SIM Confirmation of Tetrachlorodibenzo-p-dioxins

Selected flue gas and ESP ash extracts were also analyzed by HRGC/MS-SIM and HRGC/HRMS-SIM using an SP-2340 column to quantitate the 2,3,7,8-tetrachloro congener and to confirm identifications of tetrachlorodibenzo-p-dioxins (including the 2,3,7,8-congener). The instrument and operating parameters are listed in Table 14. An SIM chromatogram for several tetrachlorodibenzo-p-dioxin congeners is shown in Figure 5 and illustrates the chromatographic resolution achieved with the SP-2340 column.

TABLE 13. INSTRUMENT AND OPERATING PARAMETERS FOR HRGC/MS-SIM ANALYSES OF PCDDs/PCDFs

Instrument	Finnigan MAT 311-A/Incos
Column	15-m fused silica, wall-coated with DB-5
Column temperature	80°C hold 2 min, then to 325°C at 10°C/min
Carrier gas	helium at 2.5 psi
Injector	J&W on-column (1-μL injection)
Mass resolution	∿ 1,000 (m/Δm, 10% valley)

Ions measured

No. of chlorines	Dioxins (m/e)	Furans (m/e)	PFK (reference)
1	218.0/220.0	202.0/204.0	
2	252.0/254.0	242.0/244.0	242.9
3	285.9/287.9	269.9/271.9	
4	319.9/321.9	303.9/305.9	331.0
5	353.9/355.9	337.9/339.9	
6	389.8/391.8	373.8/375.8	380.8
7	423.8/425.8	407.8/409.8	
8	457.7/459.7	441.7/443.7	430.7

TABLE 14. INSTRUMENT AND OPERATING PARAMETERS FOR HRGC/HRMS-SIM ANALYSIS OF SELECTED EXTRACTS FOR 2,3,7,8-TETRACHLORODIBENZO-p-DIOXIN

Instrument	
Low resolution	Varian MAT CH-4B/Incos operated at ~ 1,000 resolution (m/Δm, 10% valley)
High resolution	Varian MAT 311-A/Incos operated at ~ 8,000 resolution (m/Δm, 10% valley)
Column	60-m fused silica, wall coated with SP-2340
Column temperature	100°C hold 4 min, then to 240°C at 25°C/min
Carrier gas	helium at 2.5 psi
Injector	J&W on-column (1-µl injection)
Ions measured	319.8967 321.8937 327,8847 ($^{37}Cl_4$-labeled surrogate) 331,9370 ($^{13}C_{12}$-labeled internal standard)
Reference ion	330.9793 (PFK)

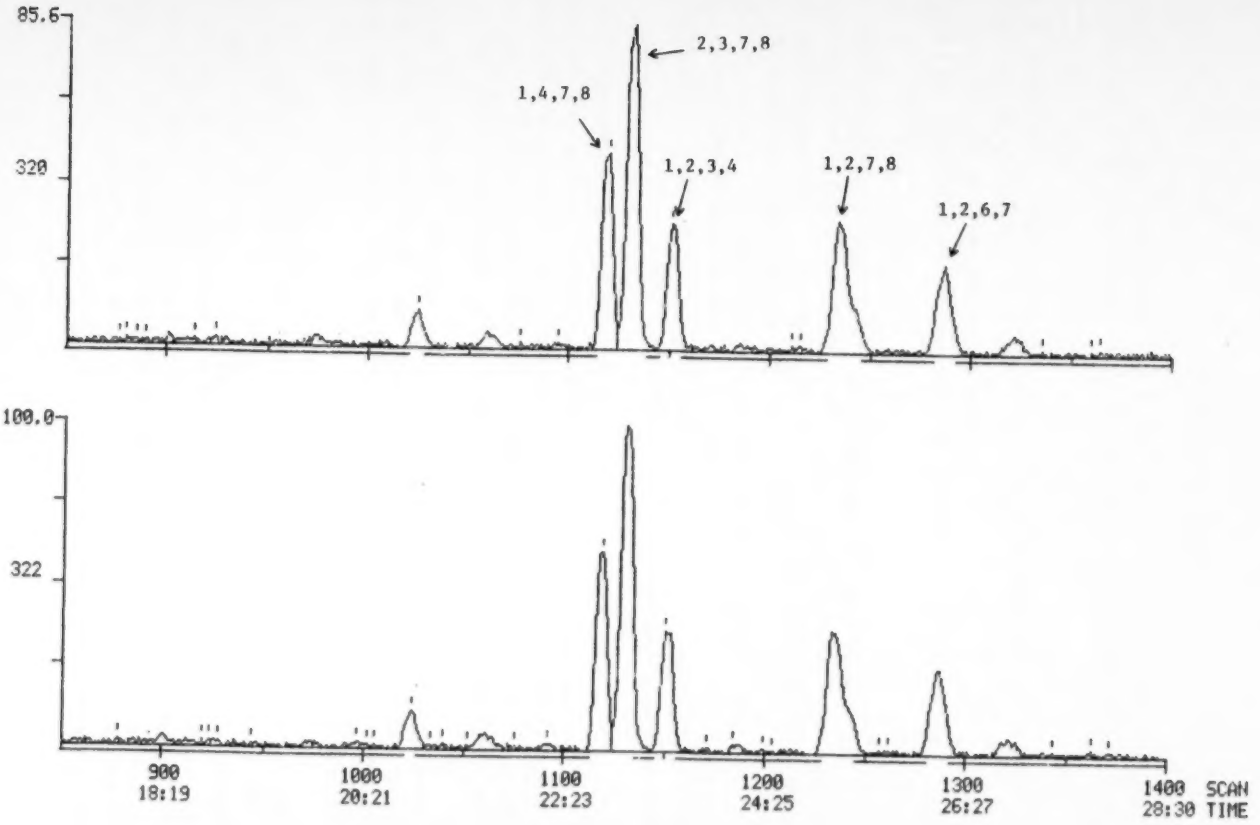

Figure 5. HRGC/MS-SIM chromatogram of tetrachlorodibenzo-p-dioxin congeners on an SP-2340 column.

SECTION 7

FIELD TEST DATA

This section presents summaries of the flue gas sampling data, unit operating parameters, and particulate control device operating data for the refuse incinerator.

A summary of the daily data for flue gas sampling as calculated from the field data sheets is presented in Table 15. The data listed are corrected to standard conditions, i.e., 20°C and a barometric pressure of 29.92 in. (1.0 atm) of mercury. Table 16 is a summary of the plant background air sampling data.

Plant operating conditions (temperatures and steam flows) and the continuous combustion gas analysis results (O_2, CO_2, CO, and THC) are plotted for the flue gas sampling periods in Figures 6 through 15. The operating temperatures are generally fairly stable during the flue gas sampling period. The furnace temperature was somewhat more variable than the ESP outlet temperature, especially on Days 2 and 3 (Figures 8 and 10). The steam flows were somewhat variable around the design steam flow of 32,000 lb/hr on Days 1 through 4. The steam flow was down somewhat on Day 5 (Figure 14).

The results of continuous monitoring of combustion gas composition were quite variable. The ranges observed were approximately 2-14% for oxygen, 6-16% for carbon dioxide, 100-3,000 ppm for carbon monoxide, and up to 300 ppm for total hydrocarbons. The patterns of changes for CO_2, CO, and THC were very similar. Oxygen followed the same patterns in the inverse direction.

Table 17 summarizes ESP operating conditions during flue gas testing. Secondary voltage (kV), secondary current (ma), and spark rate (pulses/min) were taken from ESP control panels. The secondary tier was not sparking normally during the Day 5 test.

The results of proximate and ultimate fuels analysis on ashes are shown in Table 18. On a dry basis, the bottom ash contained a larger fraction of refractory material (ash content) than the fly ash. The fly ash samples contained more volatiles, fixed carbon, and sulfur than the bottom ash and had correspondingly higher heats of combustion on a dry basis. The fly ash also contained more total chlorine. The large difference between the ash and moisture free heat of combustion of the two bottom ash composites reflects the corresponding difference in ash content.

TABLE 15. SUMMARY OF DAILY AVERAGE DATA

Test no.	Sampling location	Sample volume		Gas composition				Stack temperature (°F)	Dry molecular weight	Moisture (%)	Velocity (ft/sec)	Flue gas flow			Isokinetic rate (%)
		dscf	dscm	O_2 (%)	CO_2 (%)	CO (ppm)	THC (ppm)					acfm	dscfm	dscmm	
1	A	171.28	4.85	6.0	13.2	1,120	56.7	530	30.40	17.4	37.52	28,288	12,517	354	97.3
	B	171.45	4.86					528		18.0	36.20	27,288	12,002	340	97.6
2	A	262.53	7.43	5.5	13.5	1,230	71.5	513	30.36	17.1	36.80	27,744	12,538	355	99.3
	B	243.85	6.91					513		18.0	34.58	26,075	11,659	330	95.3
3	A	308.408	8.73	7.9	11.8	888.3	41.3	553	30.21	15.8	44.65	33,662	15,021	425	97.3
	B	308.460	8.73					538		15.8	42.78	32,263	14,339	406	98.0
4	A	254.063	7.19	6.5	12.7	1,451	71.5	520	30.19	17.7	38.98	29,389	13,074	370	92.1
	B	288.143	8.16					526		15.9	39.03	29,429	13,310	377	98.6
5	A	237.509	6.73	6.1	13.2	965.0	37.3	483	29.96	17.4	32.10	24,208	11,192	317	100.6
	B	242.887	6.88					489		17.3	31.72	23,916	10,987	311	100.7

TABLE 16. SUMMARY OF PLANT BACKGROUND AIR VOLUMES

Test	Volume	
	dscf	dscm
1	263.96	7.475
2	248.26	7.030
3	246.10	6.969
4	247.69	7.014
5	237.92	6.737

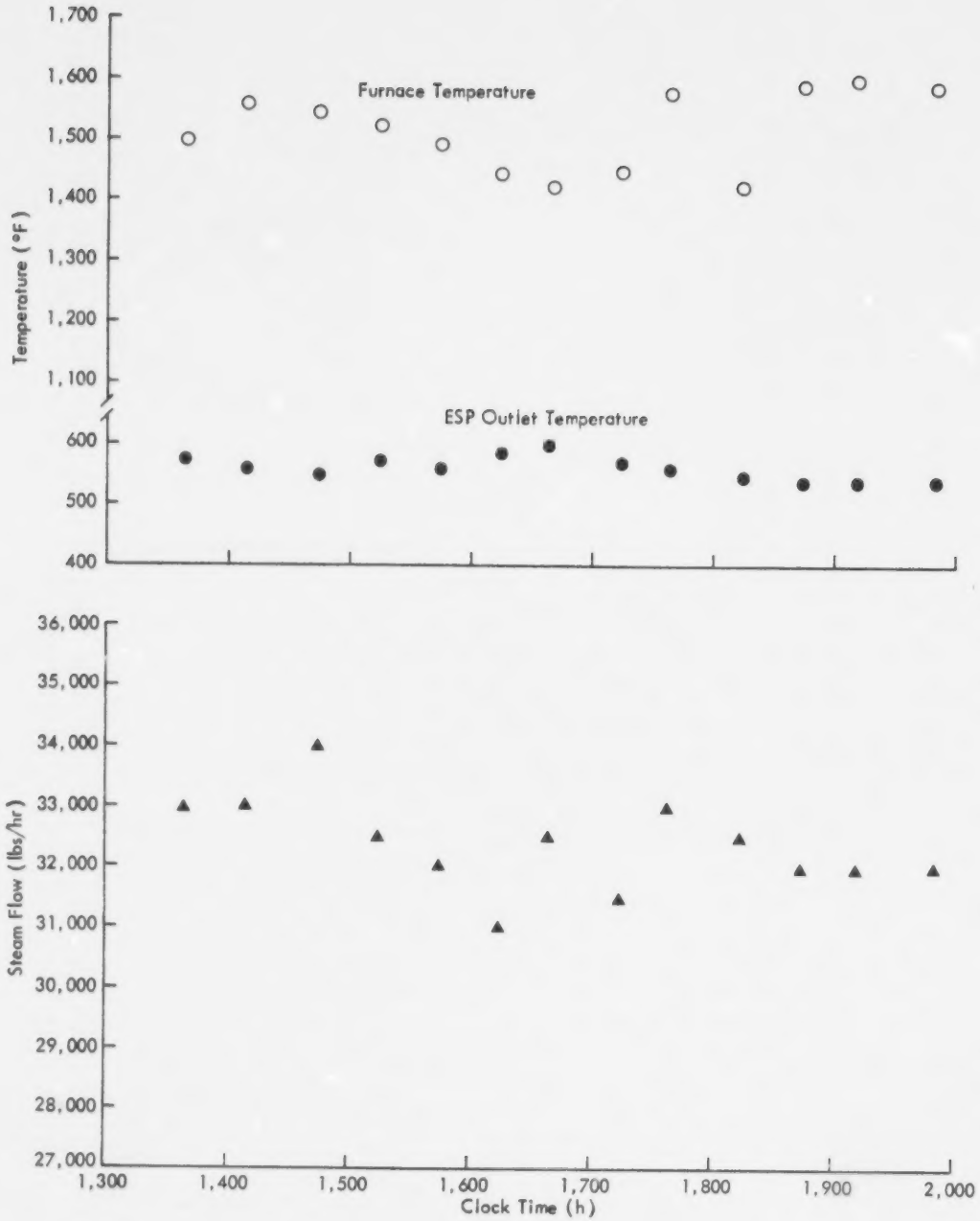

Figure 6. Operating temperatures and steam flows recorded during flue gas sampling - day 1.

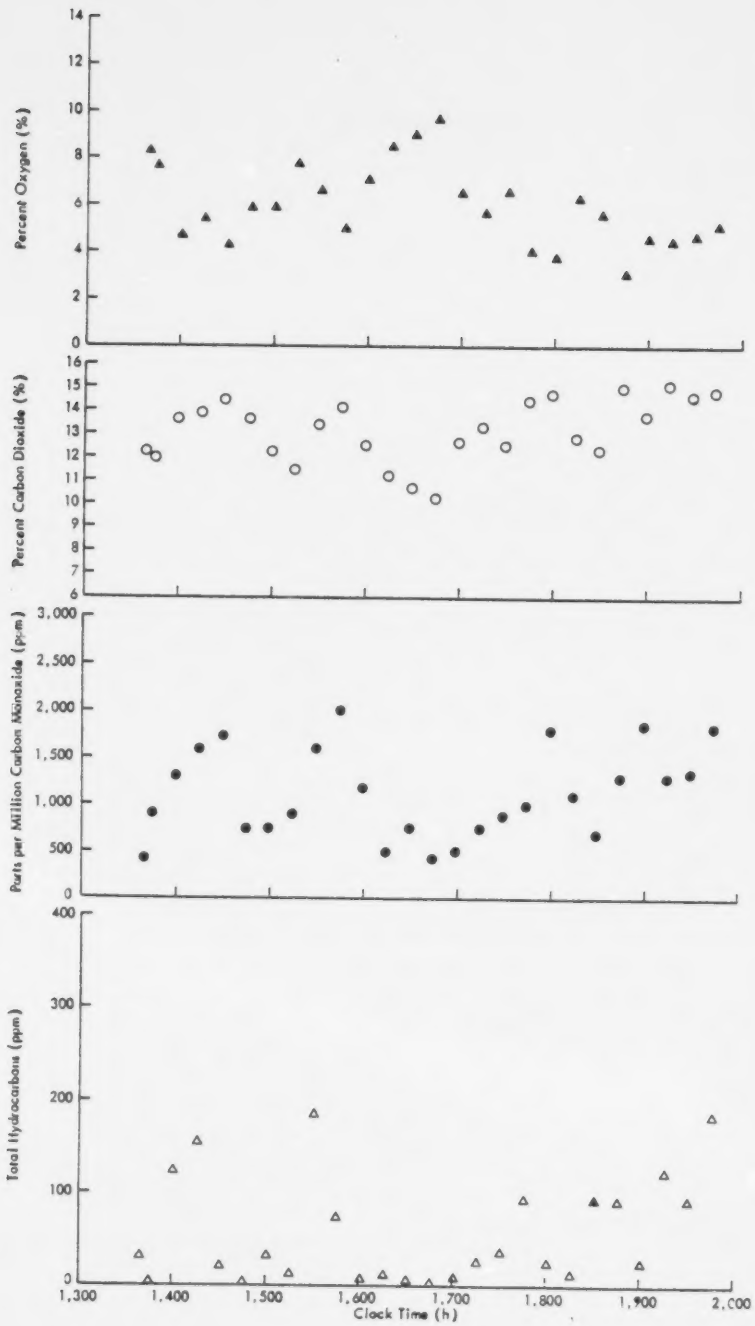

Figure 7. Combustion gas analysis results from continuous monitoring during flue gas sampling - day 1.

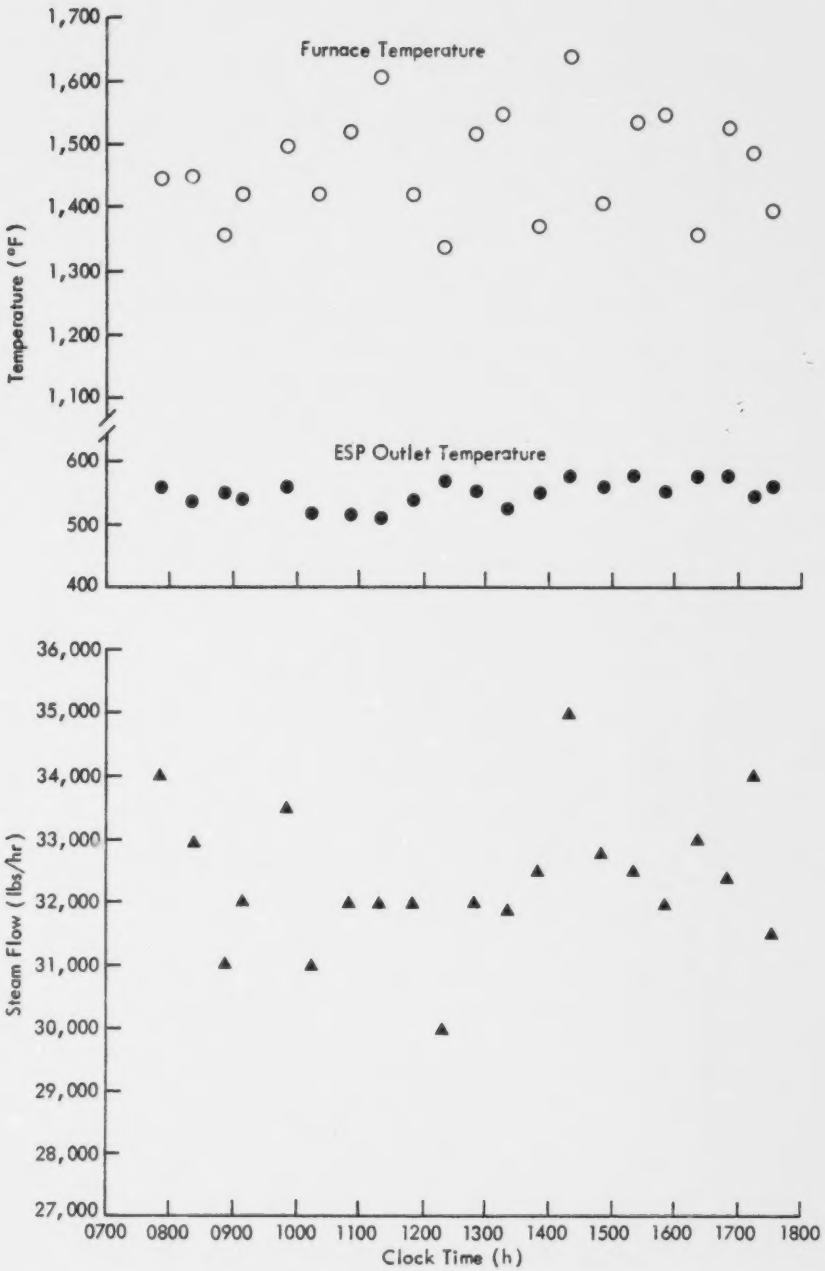

Figure 8. Operating temperatures and steam flows recorded during flue gas sampling - day 2.

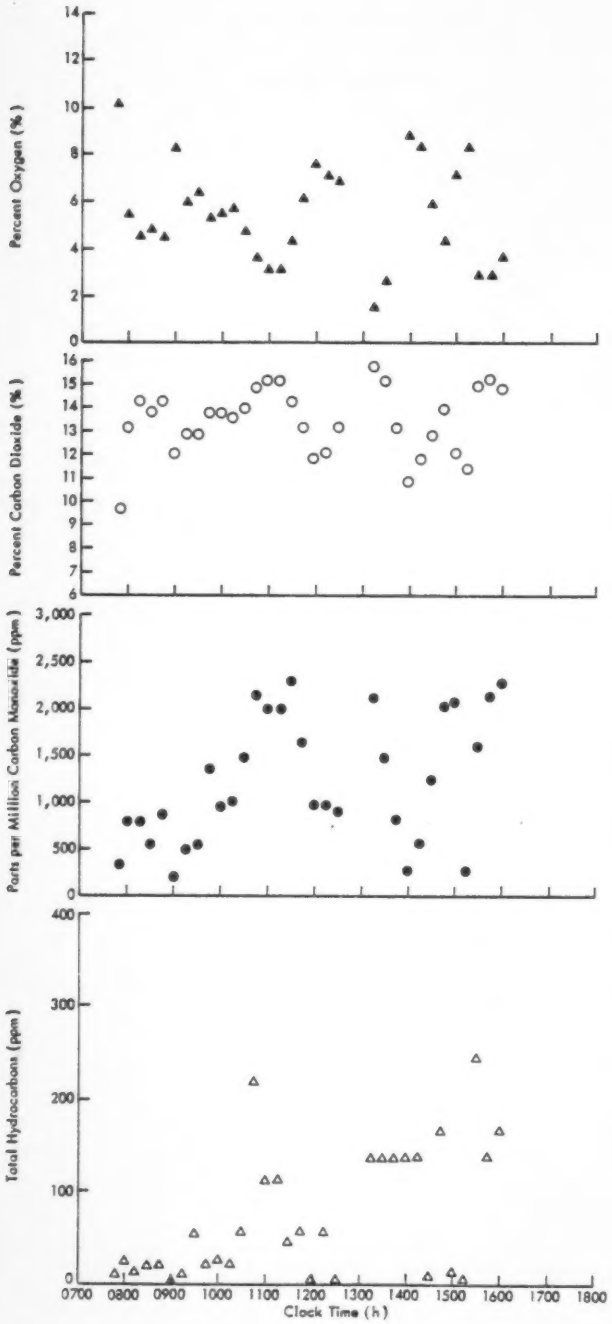

Figure 9. Combustion gas analysis results from continuous monitoring during flue gas sampling - day 2.

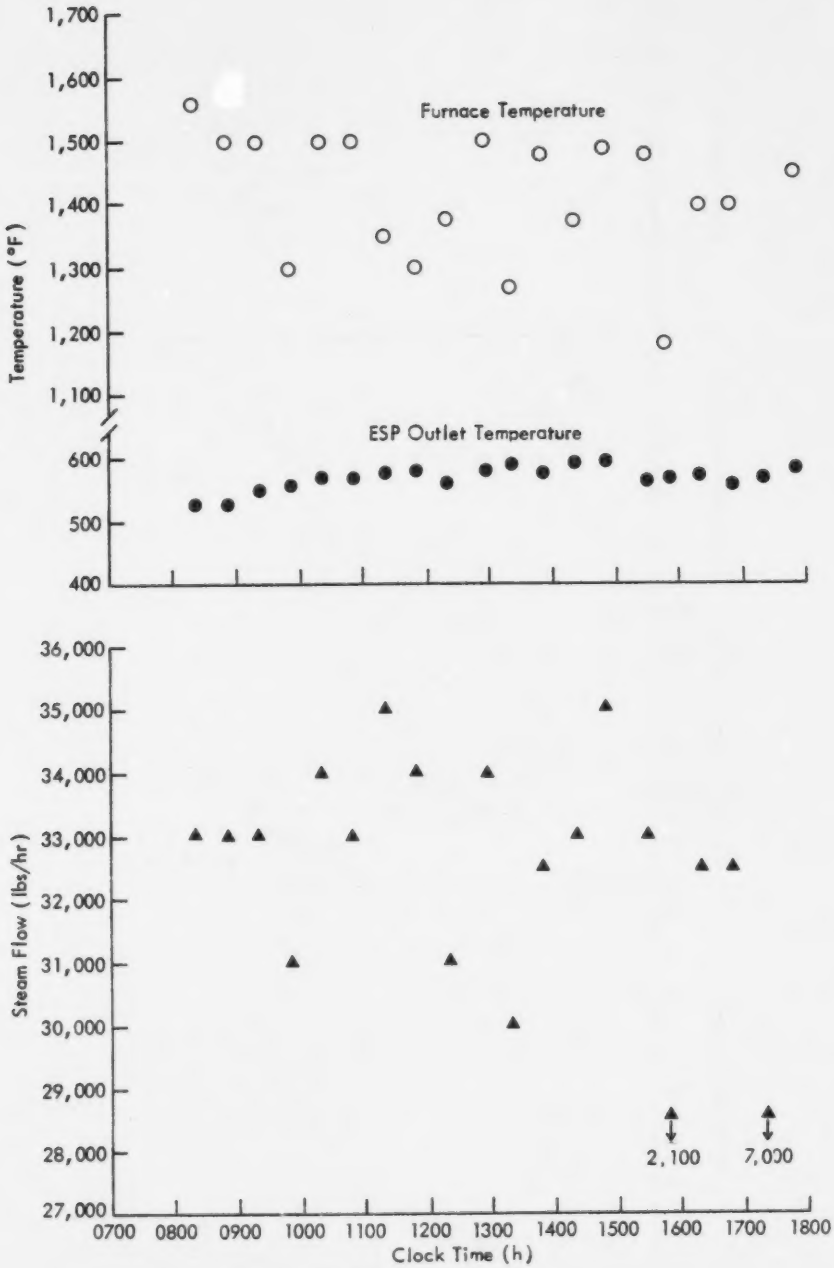

Figure 10. Operating temperatures and steam flows recorded during flue gas sampling - day 3.

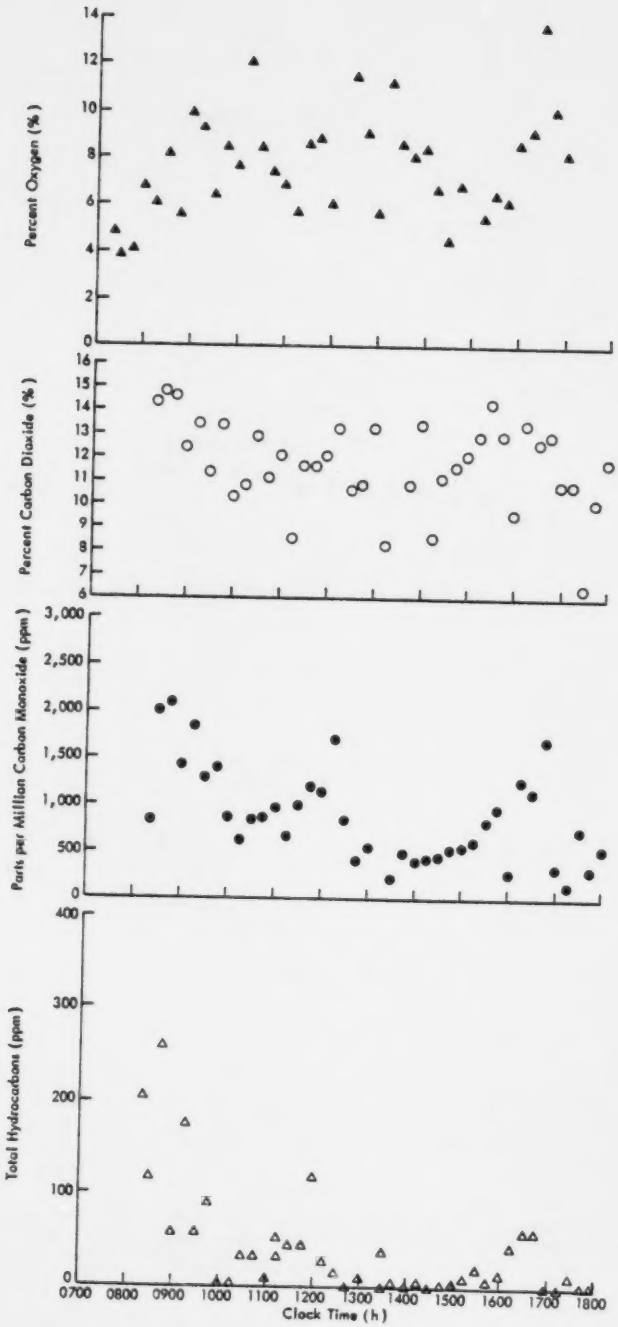

Figure 11. Combustion gas analysis results from continuous monitoring during flue gas sampling - day 3.

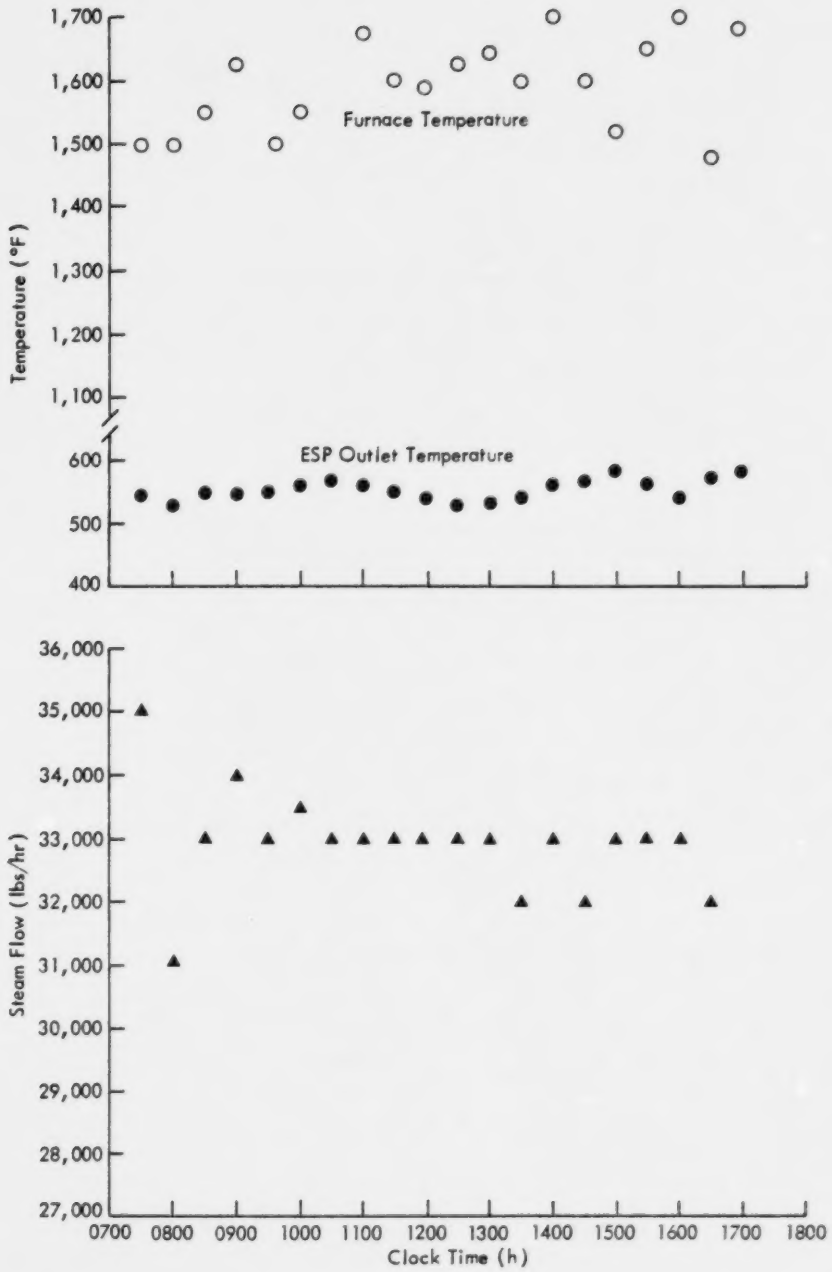

Figure 12. Operating temperatures and steam flows recorded during flue gas sampling - day 4.

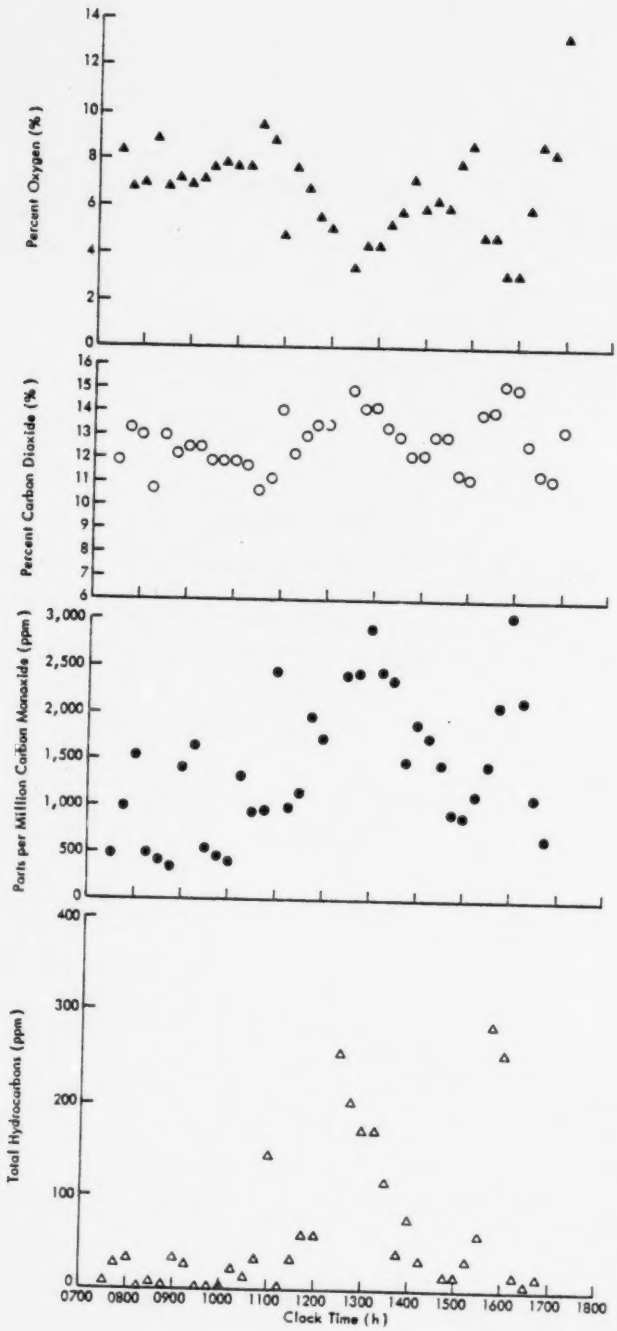

Figure 13. Combustion gas analysis results from continuous monitoring during flue gas sampling - day 4.

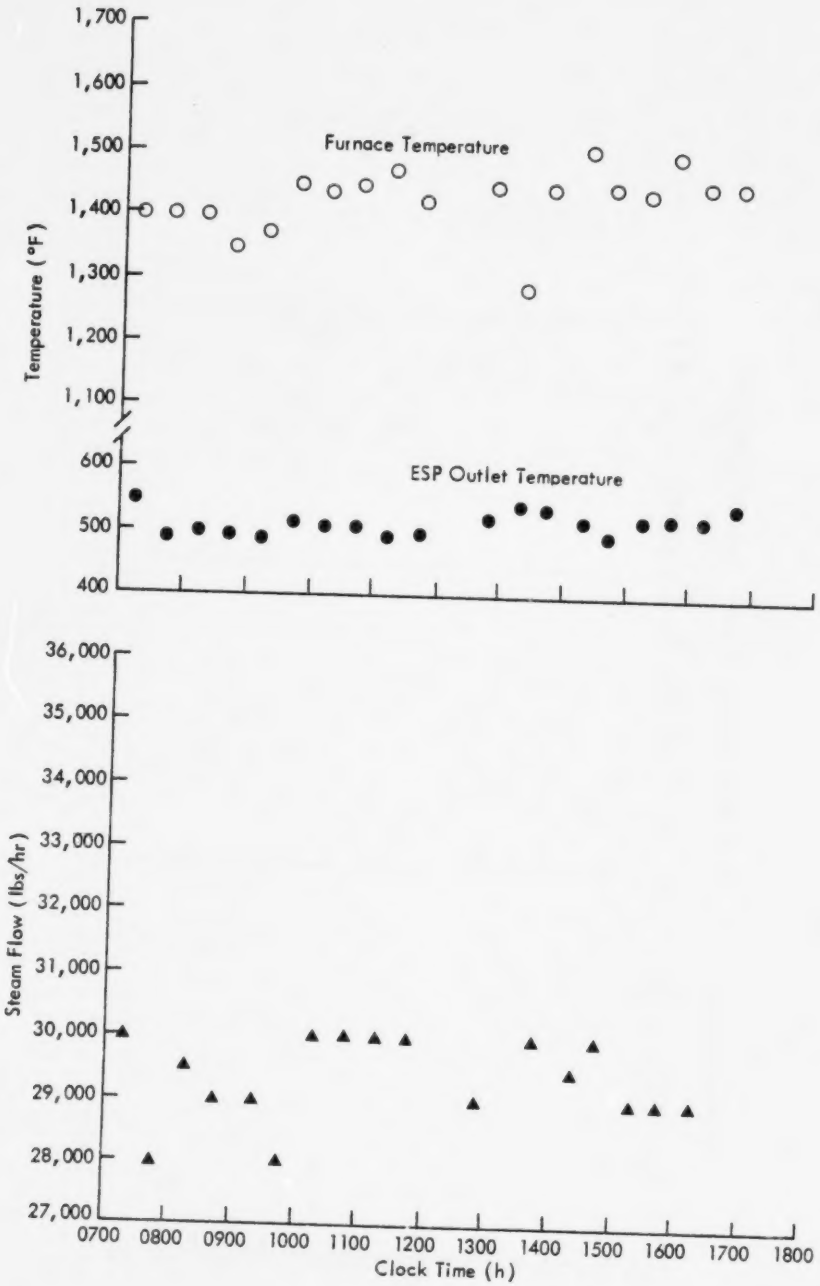

Figure 14. Operating temperatures and steam flows recorded during flue gas sampling - day 5.

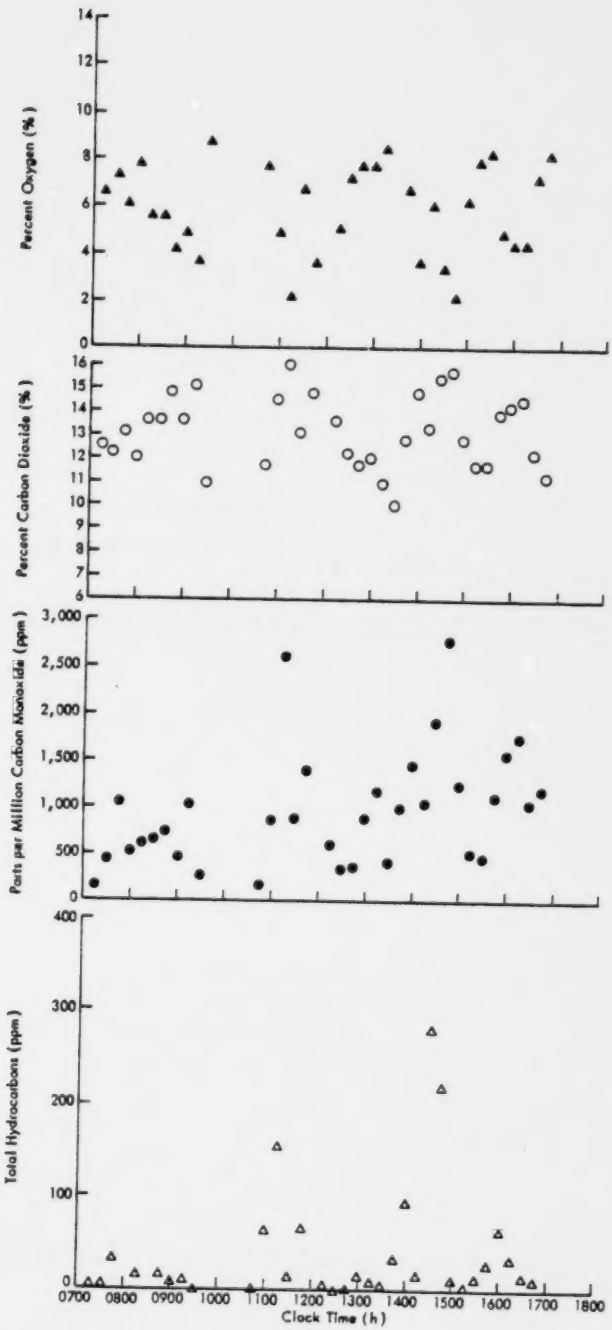

Figure 15. Combustion gas analysis results from continuous monitoring during flue gas sampling - day 5.

TABLE 17. ESP OPERATING CONDITIONS

Test day	Secondary voltage (kV)		Secondary current (ma)		Spark rate (sparks/min)	
	1° Tier	2° Tier	1° Tier	2° Tier	1° Tier	2° Tier
1 - Average	23.3	24.0	50.0	181.7	13.6	13.1
Range	24 - 24	22 - 28	40 - 60	110 - 265	4 - 49	4 - 25
2 - Average	22.0	22.1	58.5	171.5	13.9	15.0
Range	21 - 24	20 - 24	40 - 110	70 - 270	6 - 44	5 - 28
3 - Average	21.8	20.8	71.5	250	14.1	14.4
Range	21 - 23	2 - 36	45 - 190	100 - 270	6 - 25	2 - 36
4 - Average	21.8	21.9	66.5	207.5	11.5	13.8
Range	21 - 23	20 - 2.35	50 - 120	110 - 270	4 - 38	1 - 35
5 - Average	21.4	21.8	92.6	270	10.5	3.83
Range	21 - 22	21 - 23	50 - 140	200 - 280	3 - 33	0 - 14

TABLE 18. PROXIMATE AND ULTIMATE ANALYSIS RESULTS FOR BOTTOM ASH AND FLY ASH

	Bottom ash[a]	Fly ash[a]
Proximate analysis		
Reported as received		
Moisture (%)	25.68, 39.54	6.59, 4.70
Ash (%)	72.72, 53.08	73.71, 76.13
Volatile (%)	1.52, 2.77	11.70, 10.12
Fixed carbon (%)	0.08, 4.62	7.99, 9.05
Sulfur (%)	0.26, 0.35	1.21, 1.31
Heat of combustion (Btu/lb)	707, 436	1788, 1737
Dry basis		
Ash (%)	97.85, 87.79	78.92, 79.88
Volatile (%)	2.04, 4.58	12.53, 10.62
Fixed carbon (%)	0.11, 7.63	8.55, 9.50
Sulfur (%)	0.35, 0.58	1.30, 1.37
Heat of combustion (Btu/lb)	951, 721	1914, 1823
A & M free Btu (Btu/lb)	44,171, 5907	9079, 9060
Ultimate analysis		
Dry basis		
Hydrogen (%)	0.01, 0.01	0.01, 0.01
Carbon (%)	5.71, 5.98	13.90, 9.41
Nitrogen (%)	0.26, 0.29	1.05, 0.21
Oxygen (%)	4.18, 5.36	4.83, 9.12
Total chlorine (%)	0.140, 0.202 0.259, 0.238	3.926, 4.137 4.074, 3.687

a Results from two plant composite samples.

SECTION 8

ANALYTICAL RESULTS

The analytical results from this study include determinations of target PAH and phthalate compounds by HRGC/MS and PCBs, PCDDs, and PCDFs by HRGC/MS-SIM.

TARGET PAH AND PHTHALATES

The results for the target PAH and phthalate compounds identified in the flue gas samples are shown in Table 19. These data and all other analytical results reported in this document are presented without correction for recoveries. The recoveries for surrogate spikes (presented in Section 9) were generally good so that correction for the recoveries would not significantly change interpretation of the results. The results presented in Table 19 show target compound concentrations in the flue gas samples attributed to the two fractions from the sampling train, i.e., the probe rinse with associated particulate catch and the resin cartridge, as well as the sum, i.e., total, concentrations. However, the fractions of the total concentration measured for the two components should not be taken to represent particulate and vaporous fractions of the analytes. Reliable determination of particulate and vaporous fractions for many organics in flue gases are probably not possible where particulates are collected on a heated filter. Some portion of materials that enter the sampling system on particulates may subsequently vaporize from the filter deposit, held at ~ 145°C with a sampling flow near 20 L/min. Alternatively, some portion of materials that enter the sampling system as gases may be adsorbed by chemically active sites on carbonaceous or other solids deposited on the filter.

Nevertheless, all of the target PAH compounds were detected in at least one of the flue gas samples. Naphthalene, acenaphthylene, phenanthrene, fluoranthene, and pyrene concentrations exceeded 100 µg/dscm in all five flue gas samples. Phthalate concentrations were generally variable and low.

The results for target PAH and phthalate compounds in fly ash and bottom ash samples are shown in Table 20. Bottom ash included ESP and economizer ash in addition to unburned residue from both units. In general, PAH concentrations were higher in the fly ash samples and phthalate concentrations were higher in the bottom ash.

TABLE 19. TARGET COMPOUNDS IDENTIFIED IN FLUE GAS SAMPLES

Compound	Train component	Concentration (μg/dscm)					
		Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Naphthalene	Probe + filter	34	13	11	17	2.2	16 ± 12
	Resin	600	460	370	1,020	560	600 ± 250
	Sum	640	480	380	1,040	560	620 ± 250
Acenaphthylene	Probe + filter	14	19	3.0	35	9.9	16 ± 12
	Resin	200	230	110	290	170	200 ± 68
	Sum	220	250	120	330	180	220 ± 79
Acenaphthene	Probe + filter	ND[b]	ND	ND	0.35	ND	0.07 ± 0.16
	Resin	ND	3.0	1.7	5.4	2.4	2.5 ± 2.0
	Sum	ND	3.0	1.7	5.7	2.4	2.6 ± 2.1
Fluorene	Probe + filter	0.37	2.3	0.59	5.1	4.2	2.5 ± 2.1
	Resin	13	12	8.2	18	11	12 ± 3.8
	Sum	13	14	8.8	24	15	15 ± 5.4
Diethyl phthalate	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	2.5	ND	ND	1.8	1.5	1.2 ± 1.1
	Sum	2.5	ND	ND	1.8	1.5	1.2 ± 1.1
Phenanthrene	Probe + filter	11	130	55	150	130	95 ± 59
	Resin	140	88	71	99	72	93 ± 27
	Sum	150	220	130	250	200	190 ± 51
Di-n-butyl phthalate	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	12	9.7	3.5	6.2	20	10 ± 6.3
	Sum	12	9.7	3.5	6.2	20	10 ± 6.3
Fluoranthene	Probe + filter	7.4	120	43	93	110	75 ± 48
	Resin	98	45	35	38	20	47 ± 30
	Sum	110	160	78	130	130	122 ± 32
Pyrene	Probe + filter	9.1	180	46	120	120	94 ± 67
	Resin	110	55	38	40	19	53 ± 35
	Sum	120	230	84	160	140	150 ± 56

(continued)

TABLE 19 (concluded)

Compound	Train component	Concentration (μg/dscm)					
		Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Butylbenzyl phthalate	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	0.74	ND	0.84	ND	ND	0.32 ± 0.43
	Sum	0.74	ND	0.84	ND	ND	0.32 ± 0.43
Chrysene	Probe + filter	ND	9.9	4.6	14	24	11 ± 9.4
	Resin	7.7	2.0	2.4	2.8	1.2	3.2 ± 2.6
	Sum	7.7	12	7.0	17	26	14 ± 7.7
Bis[2-ethylhexyl]phthalate	Probe + filter	1.1	34	0.56	0.37	1.4	7.5 ± 15
	Resin	17	ND	0.31	2.9	6.2	5.2 ± 6.8
	Sum	18	34	0.87	3.3	7.6	13 ± 14
Benzo[k]fluoranthene	Probe + filter	1.7	15	6.1	20	26	14 ± 10
	Resin	97	1.9	2.5	3.7	1.1	21 ± 42
	Sum	99	16	8.6	23	27	35 ± 36
Benzo[a]pyrene	Probe + filter	1.4	11	4.7	13	18	9.5 ± 6.6
	Resin	6.0	1.2	1.2	1.8	0.50	2.2 ± 2.2
	Sum	7.4	12	5.9	14	19	12 ± 5.2
Dibenz[a,h]anthracene	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	ND	ND	ND	ND	0.14	0.027 ± 0.061
	Sum	ND	ND	ND	ND	0.14	0.027 ± 0.061
Benzo[g,h,i]perylene	Probe + filter	4.3	ND	ND	ND	24	4.8 ± 11
	Resin	4.3	ND	1.3	ND	1.1	1.3 ± 1.8
	Sum	4.3	ND	1.3	ND	25	6.1 ± 11

a Mean ± standard deviation for the five tests.

b Not detected, i.e., < 0.3 μg/dscm.

TABLE 20. TARGET COMPOUNDS IDENTIFIED IN ASH SAMPLES

Compound	Composite	Concentration (μg/g) Fly ash	Bottom ash[a]
Naphthalene	A	9.2	0.35
	B	9.4	0.80
	Mean	9.3	0.57
Acenaphthylene	A	3.5	0.13
	B	3.6	0.64
	Mean	3.5	0.39
Fluorene	A	0.033	ND[b]
	B	0.034	ND
	Mean	0.034	ND
Phenanthrene	A	7.8	0.27
	B	7.4	0.73
	Mean	7.6	0.50
Di-n-butyl phthalate	A	ND	0.46
	B	ND	0.26
	Mean	ND	0.36
Fluoranthene	A	6.7	0.14
	B	6.2	0.32
	Mean	6.5	0.23
Pyrene	A	5.6	0.12
	B	5.3	0.32
	Mean	5.4	0.22
Butylbenzyl phthalate	A	ND	0.084
	B	ND	0.28
	Mean	ND	0.18
Chrysene	A	0.85	ND
	B	0.54	ND
	Mean	0.69	ND
Bis[2-ethylhexyl]phthalate	A	0.17	0.56
	B	ND	3.6
	Mean	0.085	2.1
Benzo[k]fluoranthene	A	0.58	ND
	B	0.36	ND
	Mean	0.47	ND

(continued)

TABLE 20 (concluded)

Compound	Composite	Concentration (μg/g)	
		Fly ash	Bottom ash[a]
Benzo[a]pyrene	A	0.40	ND
	B	0.24	ND
	Mean	0.32	ND
Benzo[g,h,i]perylene	A	0.23	ND
	B	0.16	ND
	Mean	0.19	ND

a Includes an unknown fraction of ESP and economizer ash.

b Not detected, i.e., < 0.3 μg/g.

The results for target compounds in the aqueous and background air samples are shown in Tables 21 and 22, respectively. Although most of the target PAH compounds were identified in the quench influent and effluent samples, the concentrations observed were low. The concentrations of target compounds in the plant background air were quite variable. In general, higher levels were found in samples from Days 1 and 2. The source of compounds identified in the background air may be attributable, at least in part, to flue gas leaking from the ductwork of either or both units and collecting in the plant building. Smoke and haze were frequently observed on the upper floors during sample collection.

PCBs

The concentrations of PCB homologs identified in the flue gas samples are shown in Table 23. In general, PCB concentrations in the flue gas samples were variable from day to day. The most abundant PCB homologs were dichloro and trichloro compounds. This distribution is similar to that observed in a previous study of a mass burn municipal refuse incinerator, although the concentrations were ∿ 16 times higher in the present study.[1]

The results for PCBs in ash samples are shown in Table 24. Fly ash contained significantly higher concentrations of PCB homologs than bottom ash, although the distribution of homologs identified were similar for both sample types and also corresponded well with the homolog distribution observed for flue gas. In general, similar concentrations were observed for specific homologs in the two composites for each sample type.

PCBs were not identified in any of the aqueous or background air samples.

PCDDs

The concentrations of PCDD homologs identified in the flue gas samples are shown in Table 25. Although the concentrations varied considerably from day to day, all tetra- through octachloro homologs were identified in at least one component of the sample for each day. The total PCDD levels were very similar to concentrations reported for an incinerator of a similar design[8] but much higher than observed for the Chicago Northwest unit.[1] All homologs were identified. However, the homolog distribution maximized at pentachloro compounds with considerable contribution from tetra-, hexa-, and heptachloro-homologs. The average total PCDD concentration was 2,300 ng/dscm.

Table 26 shows the concentrations of 2,3,7,8-tetrachlorodibenzo-p-dioxin in the selected flue gas samples. The total concentration in the Day 2 sample was 8.7 ng/dscm. The fraction of the total tetrachloro homolog attributable to the 2,3,7,8- congener was 21% for the Day 2 sample and averaged 14% for all four sample components. These fractions are somewhat higher than the corresponding fraction, 6.5%, reported for the Chicago Northwest incinerator.[1]

TABLE 21. TARGET COMPOUNDS IDENTIFIED IN AQUEOUS SAMPLES

Compound	Composite	Concentration (µg/L) Influent water	Effluent water
Naphthalene	A	ND[a]	1.6
	B	2.1	3.0
	Mean	1.0	2.3
Acenaphthylene	A	28	0.46
	B	0.69	1.9
	Mean	14	1.2
Dimethyl phthalate	A	14	ND
	B	ND	ND
	Mean	7.0	ND
Fluorene	A	ND	ND
	B	ND	0.33
	Mean	ND	0.22
Diethyl phthalate	A	0.82	ND
	B	1.8	ND
	Mean	1.3	ND
Phenanthrene	A	ND	0.68
	B	2.2	5.0
	Mean	1.3	2.8
Di-n-butyl phthalate	A	13	ND
	B	0.97	ND
	Mean	6.9	ND
Fluoranthene	A	0.63	0.45
	B	1.0	1.5
	Mean	0.82	0.98
Pyrene	A	4.4	0.56
	B	1.1	1.7
	Mean	2.8	1.1
Butylbenzyl phthalate	A	5.4	ND
	B	2.3	ND
	Mean	3.9	ND
Chrysene	A	ND	ND
	B	ND	ND
	Mean	ND	ND

(continued)

TABLE 21 (concluded)

Compound	Composite	Concentration (µg/L) Influent water	Effluent water
Bis[2-ethylhexyl]phthalate	A	ND	1.8
	B	1.5	1.7
	Mean	1.0	1.8
Di-n-octyl phthalate	A	6.2	ND
	B	ND	ND
	Mean	3.2	ND

a Not detected, i.e., < 0.6 µg/L.

TABLE 22. TARGET COMPOUNDS IDENTIFIED IN PLANT BACKGROUND AIR

Compound	Concentration (ng/dscm)					
	Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Naphthalene	8,900	4,400	ND[b]	ND	ND	2,700 ± 4,000
Acenaphthylene	1,200	2,000	ND	ND	ND	640 ± 920
Acenaphthene	ND	260	ND	ND	ND	52 ± 120
Fluorene	280	87	ND	ND	ND	73 ± 120
Phenanthrene	920	780	ND	200	ND	380 ± 440
Fluoranthene	290	180	ND	120	ND	120 ± 120
Pyrene	340	190	ND	120	ND	130 ± 140
Butylbenzyl phthalate	290	110	73	170	160	160 ± 82
Chrysene	ND	74	ND	68	ND	37 ± 36
Bis[2-ethylhexyl]phthalate	1,400	790	990	370	460	800 ± 420
Benzo[k]fluoranthene	140	ND	ND	ND	ND	33 ± 61
Benzo[a]pyrene	120	ND	ND	ND	ND	24 ± 54
Dibenz[a,h]anthracene	ND	ND	ND	ND	ND	3.0 ± 6.7
Benzo[g,h,i]perylene	89	ND	ND	ND	ND	18 ± 40

a Mean ± standard deviation for the five tests.

b Not detected, i.e., < 60 ng/dscm.

TABLE 23. POLYCHLORINATED BIPHENYLS IDENTIFIED IN FLUE GAS SAMPLES

Homolog	Train component	Concentration (ng/dscm)					
		Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Monochlorobiphenyl	Probe + filter	ND[b]	21	14	45	6.3	17 ± 17
	Resin	ND	160	190	190	85	120 ± 81
	Sum	ND	180	200	230	91	140 ± 94
Dichlorobiphenyl	Probe + filter	71	150	23	370	36	130 ± 140
	Resin	ND	550	180	150	260	230 ± 210
	Sum	71	700	200	520	300	360 ± 250
Trichlorobiphenyl	Probe = filter	ND	76	150	53	43	65 ± 57
	Resin	ND	58	170	28	17	55 ± 67
	Sum	ND	130	320	81	60	120 ± 120
Tetrachlorobiphenyl	Probe + filter	ND	ND	27	18	ND	9.0 ± 13
	Resin	ND	25	34	ND	2.2	12 ± 16
	Sum	ND	25	61	18	2.2	21 ± 24
Pentachlorobiphenyl	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	56	0.8	11	19	ND	17 ± 23
	Sum	56	0.8	11	19	ND	17 ± 23
Hexachlorobiphenyl	Probe + filter	ND	13	ND	ND	ND	2.6 ± 5.7
	Resin	ND	0.5	6.7	26	ND	6.7 ± 11
	Sum	ND	13	6.7	26	ND	9.3 ± 11
Heptachlorobiphenyl	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	ND	ND	ND	0.7	ND	0.15 ± 0.33
	Sum	ND	ND	ND	0.7	ND	0.15 ± 0.33
Octachlorobiphenyl	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	ND	ND	ND	ND	ND	ND
	Sum	ND	ND	ND	ND	ND	ND

(continued)

TABLE 23 (concluded)

Homolog	Train component	Concentration (ng/dscm)					
		Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Nonachlorobiphenyl	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	ND	ND	ND	ND	ND	ND
	Sum	ND	ND	ND	ND	ND	ND
Decachlorobiphenyl	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	ND	ND	ND	ND	ND	ND
	Sum	ND	ND	ND	ND	ND	ND
Total Chlorobiphenyl	Probe + filter	71	260	220	480	86	220 ± 170
	Resin	56	800	580	420	370	440 ± 270
	Sum	130	1,100	800	900	450	670 ± 370

a Mean ± standard deviation for the five tests.

b Not detected, i.e., < 0.5 ng/dscm.

TABLE 24. POLYCHLORINATED BIPHENYLS IDENTIFIED IN ASH SAMPLES

Homolog	Composite	Concentration (ng/g) Bottom ash[a]	Fly ash
Monochlorobiphenyl	A	1.4	9.3
	B	1.3	9.7
	Mean	1.3	9.5
Dichlorobiphenyl	A	ND[b]	10
	B	ND	9.7
	Mean	ND	9.9
Trichlorobiphenyl	A	0.23	11
	B	ND	11
	Mean	0.12	11
Tetrachlorobiphenyl	A	ND	1.7
	B	ND	6.0
	Mean	ND	3.8
Pentachlorobiphenyl	A	ND	10
	B	ND	ND
	Mean	ND	5.1
Hexachlorobiphenyl	A	ND	ND
	B	ND	0.89
	Mean	ND	0.45
Heptachlorobiphenyl	A	ND	0.20
	B	ND	ND
	Mean	ND	0.10
Octachlorobiphenyl	A	ND	2.5
	B	ND	ND
	Mean	ND	1.2
Nonachlorobiphenyl	A	ND	ND
	B	ND	ND
	Mean	ND	ND
Decachlorobiphenyl	A	ND	ND
	B	ND	ND
	Mean	ND	ND
Total chlorobiphenyl	A	1.7	45
	B	1.3	37
	Mean	1.5	41

a Includes an unknown fraction of economizer and fly ash.
b Not detected, i.e., < 0.5 ng/g.

TABLE 25. POLYCHLORINATED DIBENZO-p-DIOXINS IDENTIFIED IN FLUE GAS SAMPLES

Compound	Train component	Concentration (ng/dscm)					
		Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Monodichlorodibenzo-p-dioxin	Probe + filter	ND[b]	ND	0.91	9.1	ND	2.0 ± 4.0
	Resin	13	6.5	4.0	3.2	ND	5.4 ± 5.0
	Sum	13	6.5	4.9	12	ND	7.4 ± 5.5
Dichlorodibenzo-p-dioxin	Probe + filter	ND	ND	38	130	ND	34 ± 57
	Resin	26	ND	ND	ND	ND	5.3 ± 12
	Sum	26	ND	38	130	ND	39 ± 54
Trichlorodibenzo-p-dioxin	Probe + filter	ND	ND	70	140	20	45 ± 58
	Resin	ND	ND	ND	ND	ND	ND
	Sum	ND	ND	70	140	20	45 ± 58
Tetrachlorodibenzo-p-dioxin	Probe + filter	75	34	450	370	110	210 ± 190
	Resin	88	8.0	5.2	ND	ND	20 ± 38
	Sum	160	42	450	370	110	230 ± 180
Pentachlorodibenzo-p-dioxin	Probe + filter	660	270	2,800	1,500	480	1,200 ± 1,100
	Resin	420	ND	ND	ND	ND	84 ± 190
	Sum	1,100	270	2,800	1,500	480	1,200 ± 1,000
Hexachlorodibenzo-p-dioxin	Probe + filter	430	230	770	540	160	430 ± 250
	Resin	310	21	28	43	6.0	81 ± 130
	Sum	730	250	800	590	160	510 ± 280
Heptachlorodibenzo-p-dioxin	Probe + filter	190	86	210	150	40	130 ± 70
	Resin	85	4.2	5.2	17	2.2	23 ± 35
	Sum	270	91	210	170	42	160 ± 92
Octachlorodibenzo-p-dioxin	Probe + filter	63	20	35	32	14	33 ± 19
	Resin	30	0.62	1.2	6.8	0.84	7.9 ± 13
	Sum	93	21	36	39	15	41 ± 31
Total chlorodibenzo-p-dioxins	Probe + filter	1,400	650	4,400	2,900	820	2,040 ± 1,600
	Resin	970	41	43	70	9.1	230 ± 410
	Sum	2,400	690	4,500	3,000	830	2,300 ± 1,600

a Mean ± standard deviation for the five tests.
b Not detected, i.e., < 0.5 ng/dscm.

TABLE 26. CONCENTRATIONS OF 2,3,7,8-TETRACHLORODIBENZO-p-DIOXIN IN SELECTED FLUE GAS SAMPLES

Sample	Train component	Concentration (ng/dscm)	Fraction of Total Tetrachlorodibenzo-p-dioxin (%)
Day 1	Resin	12	14
Day 2	Probe + filter	7.2	21
	Resin	1.5	19
	Sum	8.7	21
Day 4	Probe + filter	7.6	2.1

The results for PCDDs identified in ash samples are shown in Table 27. All homologs were identified in the fly ash composite samples. The mean total PCDD in the fly ash was 800 ng/g. The distribution of homologs in the ESP ash was very similar to that observed in the flue gas samples, i.e., largely pentachloro congeners with significant contributions from tetrachloro and hexachloro congeners. Only the tetrachloro homolog was identified in the bottom ash composite samples. The very low concentrations found in the bottom ash may reflect the contribution of ESP ash to the bottom ash samples.

The concentrations of 2,3,7,8-tetrachlorodibenzo-p-dioxin identified in the two fly ash composites by HRGC/HRMS-SIM were 2.9 and 1.4 ng/g. These contributions represent 1.7 and 0.8%, respectively, of the total tetrachloro homolog concentrations. Figure 16 is a chromatogram for a fly ash extract showing the 2,3,7,8- congener.

Table 28 shows the results of analysis of the plant background air for PCDDs. The very low and variable levels of PCDD homologs identified are likely attributable, at least in part, to flue gas leaking from duct work in the plant building. PCDDs were not identified in any of the quench water influent or effluent samples.

PCDFs

The concentrations of PCDF homologs identified in the flue gas samples are shown in Table 29. Considerable variation in concentrations is apparent between days. Nonetheless, nearly all homologs were identified in all sample components for each sampling day. The average total PCDF concentration was 11,000 ng/dscm. The homolog distribution was similar to that for PCDDs in flue gas, i.e., largely pentachloro congeners. However, the average total PCDF levels were over 4 times higher than the mean total PCDD concentration. The PCDF/PCDD ratio observed in flue gas from the Chicago Northwest incinerator was over 10.

TABLE 27. POLYCHLORINATED DIBENZO-p-DIOXINS IDENTIFIED IN ASH SAMPLES

Homolog	Composite	Concentration (ng/g) Bottom ash[a]	Fly ash
Monochlorodibenzo-p-dioxin	A	ND[b]	2.5
	B	ND	1.6
	Mean	ND	2.0
Dichlorodibenzo-p-dioxin	A	ND	21
	B	ND	24
	Mean	ND	22
Trichlorodibenzo-p-dioxin	A	ND	68
	B	ND	43
	Mean	ND	55
Tetrachlorodibenzo-p-dioxin	A	0.13	170
	B	0.50	170
	Mean	0.32	170
Pentachlorodibenzo-p-dioxin	A	ND	650
	B	ND	420
	Mean	ND	530
Hexachlorodibenzo-p-dioxin	A	ND	50
	B	ND	54
	Mean	ND	52
Heptachlorodibenzo-p-dioxin	A	ND	6.3
	B	ND	8.4
	Mean	ND	7.4
Octachlorodibenzo-p-dioxin	A	ND	4.1
	B	ND	1.1
	Mean	ND	2.6
Total chlorodibenzo-p-dioxins	A	0.13	1,000
	B	0.50	720
	Mean	0.32	800

a Includes an unknown fraction of economizer and ESP ash.

b Not detected, i.e., < 0.05 ng/g.

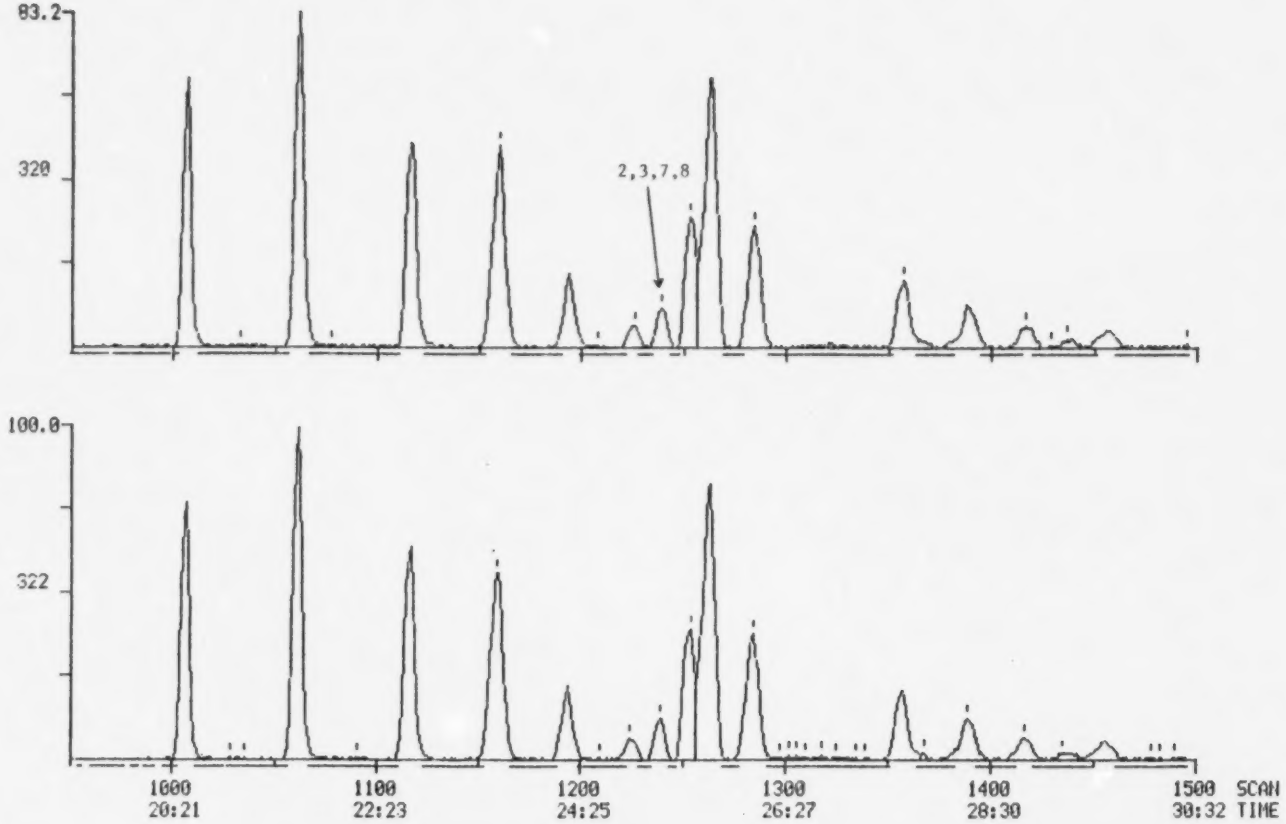

Figure 16. HRGC/HRMS-SIM chromatogram for fly ash composite A extract.

TABLE 28. POLYCHLORINATED DIBENZO-p-DIOXINS IDENTIFIED IN PLANT BACKGROUND AIR

Homolog	Concentration (ng/dscm)					
	Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Monochlorodibenzo-p-dioxin	ND[b]	ND	ND	ND	ND	ND
Dichlorodibenzo-p-dioxin	ND	ND	ND	ND	ND	ND
Trichlorodibenzo-p-dioxin	ND	ND	ND	ND	ND	ND
Tetrachlorodibenzo-p-dioxin	ND	ND	ND	0.24	ND	0.048 ± 0.11
Pentachlorodibenzo-p-dioxin	ND	ND	ND	ND	ND	ND
Hexachlorodibenzo-p-dioxin	ND	ND	ND	ND	ND	ND
Heptachlorodibenzo-p-dioxin	ND	ND	ND	5.7	ND	1.1 ± 2.5
Octachlorodibenzo-p-dioxin	0.35	ND	ND	11	ND	2.3 ± 4.9
Total chlorinated dibenzo-p-dioxins	0.35	ND	ND	17	ND	3.5 ± 7.5

a Mean ± standard deviation for the five tests.

b Not detected, i.e., < 0.15 ng/dscm.

TABLE 29. POLYCHLORINATED DIBENZOFURANS IDENTIFIED IN FLUE GAS SAMPLES

Homolog	Train component	Concentration (ng/dscm)					
		Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Monochlorodibenzofuran	Probe + filter	71	27	58	140	130	85 ± 48
	Resin	310	370	240	280	180	280 ± 72
	Sum	380	400	300	420	310	360 ± 54
Dichlorodibenzofuran	Probe + filter	100	170	290	550	390	300 ± 180
	Resin	300	310	210	140	56	210 ± 110
	Sum	400	490	500	700	440	510 ± 110
Trichlorodibenzofuran	Probe + filter	570	680	1,800	3,000	1,500	1,500 ± 980
	Resin	1,200	460	310	360	82	480 ± 430
	Sum	1,800	1,100	2,100	3,300	1,600	2,000 ± 830
Tetrachlorodibenzofuran	Probe + filter	330	350	1,900	1,400	550	910 ± 710
	Resin	470	130	110	110	9.6	170 ± 180
	Sum	800	480	2,000	1,600	560	1,100 ± 670
Pentachlorodibenzofuran	Probe + filter	1,400	1,100	15,000	8,800	2,900	5,800 ± 5,900
	Resin	1,400	200	210	350	19	430 ± 550
	Sum	2,800	1,300	15,000	9,200	2,900	6,200 ± 5,700
Hexachlorodibenzofuran	Probe + filter	ND[b]	170	1,800	920	330	650 ± 750
	Resin	210	0.15	4.4	25	5.1	49 ± 91
	Sum	210	170	1,800	950	340	700 ± 710
Heptachlorodibenzofuran	Probe + filter	140	96	370	220	80	180 ± 120
	Resin	77	6.2	6.7	14	3.6	22 ± 31
	Sum	210	100	380	230	83	200 ± 120
Octachlorodibenzofuran	Probe + filter	8	8.8	24	18	9.2	14 ± 7.2
	Resin	ND	ND	ND	ND	ND	ND
	Sum	8	8.8	24	18	9.2	14 ± 7.2
Total chlorodibenzofurans	Probe + filter	2,600	2,600	21,000	15,000	5,900	9,400 ± 8,200
	Resin	4,000	1,500	1,100	1,300	360	1,600 ± 1,400
	Sum	6,600	4,100	22,000	16,000	6,300	11,000 ± 7,700

a Mean ± standard deviation for the five tests.
b Not detected, i.e., < 0.5 ng/dscm.

Table 30 shows the results for PCDFs identified in the ash samples. All homologs were identified in the fly ash composites. As noted for PCDDs in fly ash, the distribution of PCDF homologs is similar to that for flue gas. The mean PCDF concentration in the ESP ash was 3,000 ng/g, over three times the mean PCDD level. Also, the low PCDF concentration determined in the bottom ash (mean of 9.3 ng/g) may reflect the ESP contribution to bottom ash samples.

PCDFs were also identified in the plant background air samples. The results are shown in Table 31. As noted for PCDDs, PCDF levels in background air were very low. PCDFs were not identified in any quench water samples.

TABLE 30. POLYCHLORINATED DIBENZOFURANS IDENTIFIED IN ASH SAMPLES

Homolog	Composite	Concentration (ng/g) Bottom ash[a]	Fly ash
Monochlorodibenzofuran	A	0.83	42
	B	1.4	40
	Mean	1.1	41
Dichlorodibenzofuran	A	0.38	69
	B	0.89	110
	Mean	0.63	90
Trichlorodibenzofuran	A	ND[b]	520
	B	ND	580
	Mean	ND	550
Tetrachlorodibenzofuran	A	0.88	350
	B	2.0	460
	Mean	1.4	410
Pentachlorodibenzofuran	A	3.5	1,800
	B	8.9	1,800
	Mean	6.2	1,800
Hexachlorodibenzofuran	A	ND	75
	B	ND	91
	Mean	ND	83
Heptachlorodibenzofuran	A	ND	8.5
	B	ND	11
	Mean	ND	9.5
Octachlorodibenzofuran	A	ND	2.2
	B	ND	0.57
	Mean	ND	1.4
Total chlorinated dibenzofurans	A	5.5	2,800
	B	13	3,100
	Mean	9.3	3,000

a Includes an unknown fraction of ESP and economizer ash.

b Not detected, i.e., < 0.05 ng/g.

TABLE 31. POLYCHLORINATED DIBENZOFURANS IN PLANT BACKGROUND AIR

Homolog	Concentration (ng/dscm)					
	Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Monodichlorodibenzofuran	ND[b]	ND	ND	ND	ND	ND
Dichlorodibenzofuran	ND	ND	ND	ND	ND	ND
Trichlorodibenzofuran	ND	ND	ND	ND	ND	ND
Tetrachlorodibenzofuran	1.1	ND	0.27	0.39	ND	0.35 ± 0.45
Pentachlorodibenzofuran	3.8	ND	ND	ND	ND	0.75 ± 1.7
Hexachlorodibenzofuran	ND	ND	ND	ND	ND	ND
Heptachlorodibenzofuran	ND	ND	ND	1.5	ND	0.30 ± 0.76
Octachlorodibenzofuran	ND	ND	ND	7.1	ND	1.4 ± 3.2
Total chlorinated dibenzofurans	4.8	ND	0.27	9.0	ND	2.8 ± 4.0

a Mean ± standard deviation for the five tests.

b Not detected, i.e., < 0.15 ng/dscm.

SECTION 9

ANALYTICAL QUALITY ASSURANCE RESULTS

The analytical quality assurance program included the use of surrogate spiking compounds, analysis of blank samples, and analysis of a standard reference material (dust). In addition, impinger samples were analyzed to determine analyte breakthrough during flue gas sampling. These results are presented and discussed below.

SURROGATE COMPOUND RECOVERIES

The primary indicators of the performance of the analytical procedures were the recoveries of surrogate analytes spiked into samples prior to extraction and analysis. Three classes of surrogate compounds were used for this study: stable isotope-labeled surrogates for the target compounds, stable isotope-labeled PCB congeners, and stable isotope-labeled PCDD congeners.

The recoveries of the surrogates for the target compounds spiked into flue gas samples are shown in Table 32. The corresponding recoveries for surrogates in plant background air are shown in Table 33. Table 34 presents the summary of recoveries for all samples. The recoveries and standard deviations generally indicate that the precision and accuracy were generally good for all compounds except pentachlorophenol-$^{13}C_6$. Pentachlorophenol is very polar and acidic, more polar than the target analytes. This characteristic is manifested in a gas chromatographic peak shape for PCP that is generally broad and very susceptible to changes in the activity of the column. Hence, the recovery of pentachlorophenol-$^{13}C_6$ provides an indication of the maximum apparent losses due to adsorption on the fused silica capillary column. Also, the recoveries for naphthalene-d_8 from the flue gas resin samples were somewhat variable. This may indicate some losses for very volatile analytes.

The recoveries for the surrogate polychlorinated biphenyls from flue gas and background air samples are shown in Tables 35 and 36, respectively. The summary of recoveries for all samples is presented in Table 37. Recoveries of the PCB surrogates were generally low for flue gas, fly ash, and bottom ash samples, especially in the case of the monochloro compound. This may be partially attributable to the high levels of extractable organics in these extracts. Even following cleanup, the flue gas, fly ash, and bottom ash extracts required dilution prior to analysis. Higher spike levels may have been appropriate for those samples. Nonetheless, recoveries were generally good for the background air and quench water samples.

TABLE 32. RECOVERIES OF SURROGATE TARGET COMPOUNDS SPIKED INTO THE FLUE GAS SAMPLES

Surrogate	Train component	Recovery (%)				
		Day 1	Day 2	Day 3	Day 4	Day 5
Naphthalene-d_8	Probe + filter	50	83	90	80	55
	Resin	140	29	21	68	31
Chrysene-d_{12}	Probe + filter	29	66	59	66	69
	Resin	110	80	66	100	94
1,2,4,5-Tetrachlorobenzene-$^{13}C_6$	Probe + filter	26	47	48	44	28
	Resin	83	69	58	85	80
Pentachlorophenol-$^{13}C_6$	Probe + filter	0	0	0	0	0
	Resin	0	0	0	0	0

TABLE 33. RECOVERIES OF SURROGATE TARGET COMPOUNDS SPIKED INTO THE PLANT BACKGROUND AIR SAMPLES

Surrogate	Recovery (%)				
	Day 1	Day 2	Day 3	Day 4	Day 5
Naphthalene-d_8	77	92	83	86	96
Chrysene-d_{12}	93	85	82	102	81
1,2,4,5-Tetrachlorobenzene-$^{13}C_6$	76	65	51	71	51
Pentachlorophenol-$^{13}C_6$	2.1	0	0	2.0	0.80

TABLE 34. SUMMARY OF RECOVERIES OF THE SURROGATE TARGET COMPOUNDS

Sample type	% Recovery			
	Naphthalene-d_8	Chrysene-d_{12}	1,2,4,5-Tetrachlorobenzene-$^{13}C_6$	Pentachlorophenol-$^{13}C_6$
Flue gas, probe + filter	72 ± 18[a]	58 ± 17	39 ± 11	0
Flue gas, resin	58 ± 49	90 ± 17	75 ± 11	0
Plant background air	87 ± 8	89 ± 9	63 ± 11	0.98 ± 1.0
Fly ash	80, 79[b]	64, 37	78, 80	0, 0
Bottom ash	64, 88	82, 74	72, 21	0, 0
Quench influent	60, 97	73, 98	65, 67	0, 43
Quench effluent	70, 65	68, 93	48, 73	0, 0

a Mean ± standard deviation for the five tests.

b From analysis of two spiked composites.

TABLE 35. RECOVERIES OF POLYCHLORINATED BIPHENYL SURROGATES SPIKED INTO FLUE GAS SAMPLES

Surrogate	Train component	Recovery (%)				
		Day 1	Day 2	Day 3	Day 4	Day 5
4-Chlorobiphenyl-$^{13}C_6$	Probe + filter	0	0	0	0	0
	Resin	0	0	0	0	0
3,3',4,4'-Tetrachlorobiphenyl-$^{13}C_{12}$	Probe + filter	33	72	0	17	19
	Resin	0	27	23	43	57
2,2',3,3',5,5',6,6'-Octachlorobiphenyl-$^{13}C_{12}$	Probe + filter	37	27	27	32	53
	Resin	15	45	50	23	44
Decachlorobiphenyl-$^{13}C_{12}$	Probe + filter	15	12	16	18	20
	Resin	43	30	37	34	33

TABLE 36. RECOVERIES OF POLYCHLORINATED BIPHENYL SURROGATES SPIKED INTO PLANT BACKGROUND AIR SAMPLES

Surrogate	Recovery (%)				
	Day 1	Day 2	Day 3	Day 4	Day 5
4-Chlorobiphenyl-$^{13}C_{12}$	93	0	73	82	0
3,3',4,4'-Tetrachlorobiphenyl-$^{13}C_{12}$	69	75	65	66	61
2,2',3,3',5,5',6,6'-Octachlorobiphenyl-$^{13}C_{12}$	53	73	63	73	53
Decachlorobiphenyl-$^{13}C_{12}$	55	61	70	82	30

TABLE 37. SUMMARY OF RECOVERIES OF THE POLYCHLORINATED BIPHENYL SURROGATE COMPOUNDS

Sample type	4-Chlorobiphenyl-$^{13}C_6$	3,3',4,4'-Tetrachlorobiphenyl-$^{13}C_{12}$	2,2',3,3',5,5',6,6'-Octachlorobiphenyl-$^{13}C_{12}$	Decachlorobiphenyl-$^{13}C_{12}$
Flue gas, probe + filter	0	28 ± 27[a]	35 ± 11	16 ± 3.2
Flue gas, resin	0	30 ± 22	36 ± 15	35 ± 4.9
Plant background air	45 ± 46	67 ± 5.7	63 ± 10	60 ± 19
Fly ash	0, 0[b]	0, 26	45, 45	38, 37
Bottom ash	0, 0	33, 67	37, 58	0, 48
Quench water influent	54, 58	54, 48	52, 63	46, 66
Quench water effluent	57, 67	44, 74	68, 76	150, 170

a Mean ± standard deviation for five samples.

b Determinations from two composite samples.

Tables 38 and 39 show the polychlorinated dibenzo-p-dioxin surrogate recoveries for flue gas and background air samples, respectively. Table 40 summarizes the recoveries for all samples. Recoveries were generally good for both compounds from flue gas, background air, and the quench water samples. However, the octachloro compound was inexplicably poorly recovered from ashes. Typically, low recoveries for octachlorodibenzo-p-dioxin may be attributable to poor chromatography sometimes enhanced by its long elution time.

BLANK SAMPLES

The levels of analytes determined in blank samples were not detectable or were negligible relative to concentrations determined in the corresponding samples. Table 41 shows the analytes identified in blank samples.

STANDARD REFERENCE MATERIALS

Standard Reference Material 1649, "Urban Dust/Organics" was analyzed (in duplicate) in parallel with and by the same methods used for ESP ash analysis. The National Bureau of Standards has certified concentrations for five polynuclear aromatic hydrocarbons in SRM 1649. These are fluoranthene, benz[a]anthracene, benzo[a]pyrene, benzo[g,h,i]perylene, and indeno[1,2,3-cd]pyrene. The certified values were derived from analytical results from highly specific gas chromatographic and liquid chromatographic methods. Only two of the certified compounds, benzo[g,h,i]perylene and fluoranthene, are uniquely determined by the more broadly applicable HRGC/MS method used in this study. Indeno[1,2,3-cd]pyrene was not a target analyte. Benz[a]anthracene and benzo[a]pyrene co-elute with their isomers and cannot be distinguished from them by mass spectrometry. Table 42 shows the certified values and the analysis results for the two compounds. These results indicate good correspondence with the certified values.

FLUE GAS ANALYTE BREAKTHROUGH TESTS

The results of analyses of the first impinger contents from flue gas sampling on Days 2 and 4 are shown in Table 43. These results indicate that breakthrough was only a negligible fraction of the analytes identified.

TABLE 38. RECOVERIES OF POLYCHLORINATED DIBENZO-p-DIOXIN SURROGATES SPIKED INTO FLUE GAS SAMPLES

Surrogate	Train component	Recovery (%)				
		Day 1	Day 2	Day 3	Day 4	Day 5
2,3,7,8-Tetrachlorodibenzo-p-dioxin-$^{37}Cl_4$	Probe + filter	79	53	90	77	82
	Resin	75	84	68	81	67
Octachlorodibenzo-p-dioxin-$^{13}C_{12}$	Probe + filter	98	60	65	56	51
	Resin	85	44	95	170	110

TABLE 39. RECOVERIES OF POLYCHLORINATED DIBENZO-p-DIOXIN SURROGATES SPIKED INTO PLANT BACKGROUND AIR SAMPLES

Surrogate	Recovery (%)				
	Day 1	Day 2	Day 3	Day 4	Day 5
2,3,7,8-Tetrachlorodibenzo-p-dioxin-$^{37}Cl_4$	88	74	89	83	75
Octachlorodibenzo-p-dioxin-$^{13}C_{12}$	56	37	0	120	84

TABLE 40. SUMMARY OF RECOVERIES OF THE POLYCHLORINATED DIBENZO-p-DIOXIN SURROGATE COMPOUNDS

Sample type	Recovery (%)	
	2,3,7,8-Tetrachlorodibenzo-p-dioxin-$^{37}Cl_4$	Octachlorodibenzo-p-dioxin-$^{13}C_{12}$
Flue gas, probe + filter	76 ± 14[a]	66 ± 19
Flue gas, resin	77 ± 7.5	100 ± 47
Plant background air	82 ± 7.2	59 ± 45
Fly ash	55, 45	5, 0
Bottom ash	43, 80	10, 100
Quench water influent	77, 76	59, 38
Quench water effluent	79, 88	58, 62

a Mean ± standard deviation for the five tests.

b Determinations for two composite samples.

TABLE 41. ANALYTES IDENTIFIED IN BLANK SAMPLES

Compound	Concentration[a]	
	Flue gas resin (μg/dscm)	Flue gas probe + filter (ng/dscm)
Naphthalene	0.23	0.20
Diethylphthalate	0.07	0.72
Phenanthrene	0.02	ND[b]
Di-n-butylphthalate	0.28	0.39
Fluoranthene	0.02	0.03
Pyrene	0.02	0.02
Butylbenzylphthalate	0.09	0.12
Bis[2-ethylhexyl]phthalate	0.05	ND
Di-n-octylphthalate	0.46	0.79

a Calculated assuming a typical sample size.

b Not detected.

TABLE 42. RESULTS FOR ANALYSIS OF SRM 1649

Compound	Concentration (μg/g)	
	Certified value	Analysis results
Fluoranthene	7.1 ± 0.5	6.3, 10.8[a]
Benzo[g,h,i]perylene	4.5 ± 1.1	5.3, 6.2

a Results from duplicate determinations.

TABLE 43. ANALYTES IDENTIFIED IN FLUE GAS TRAIN FIRST IMPINGERS

Compound	Concentration (μg/dscm)		Breakthrough[a] (%)	
	Day 2	Day 4	Day 2	Day 4
Naphthalene	0.65	1.0	0.1	0.1
Fluorene	ND	0.01	0	0.04
Chrysene	0.01	0.02	0.08	0.1
Benzo[a]pyrene	ND	0.01	0	0.07

a Concentration in the first impinger divided by the concentration in the total sample, expressed as percent.

SECTION 10

EMISSION RESULTS

The emission rates determined for the target PAH and phthalate compounds in flue gases for the resource recovery municipal incinerator are presented in Table 44. Emission rates were calculated from the concentrations determined in each sample (presented in Section 8) and the flue gas volume flow rates (presented in Section 7). Emission rates were not similarly derived for the ash samples because ash production rates for the furnace could not be reliably estimated.

The highest emission rates of the target compounds were determined for naphthalene (average of 13,000 mg/hr) and acenaphthylene (average of 4,700 mg/hr). Average emission rates over 1,000 mg/hr were also determined for phenanthrene, fluoranthene, and pyrene. As indicated in Section 8, the fractions of analytes found on the two principal flue gas sampling train components should not be interpreted as reliable indications of "particulate" or "vaporous" fractions. This is the case for the target compounds, PCBs, PCDDs, and PCDFs.

The emission rates for PCB compounds in flue gas are shown in Table 45. The mean total PCB emission rate was 15 ± 8.5 mg/hr. The lowest total PCB emissions were determined on Days 1 and 5 (2.6 and 5.8 mg/hr, respectively).

The emission rates for PCDDs and PCDFs in flue gas are shown in Tables 46 and 47, respectively. The average total PCDD emission rate was 51 ± 40 mg/hr. The emission rate was highest on Day 3 (110 mg/hr) and lowest on Days 2 and 5 (14 and 16 mg/hr, respectively). The relative order of total PCDF emission rates was very similar, lowest on Days 2 and 5 (83 and 120 mg/hr, respectively) and highest on Day 3 (550 mg/hr). The average total PCDF emission rate was 250 ± 200 mg/hr.

TABLE 44. EMISSION RATES FOR TARGET COMPOUNDS IN FLUE GAS

Compound	Train component	Emission rate (mg/hr)					
		Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Naphthalene	Probe + filter	700	270	280	390	41	340 ± 240
	Resin	13,000	9,500	9,200	23,000	11,000	13,000 ± 5,700
	Sum	13,000	9,800	9,400	23,000	11,000	13,000 ± 5,800
Acenaphthylene	Probe + filter	290	390	76	780	190	340 ± 270
	Resin	4,200	4,800	2,800	6,600	3,200	4,300 ± 1,500
	Sum	4,500	5,200	2,900	7,300	3,400	4,700 ± 1,800
Acenaphthene	Probe + filter	ND[b]	ND	ND	7.8	ND	1.6 ± 3.5
	Resin	ND	62	43	120	45	54 ± 43
	Sum	ND	62	43	130	45	56 ± 46
Fluorene	Probe + filter	7.7	48	15	120	78	53 ± 45
	Resin	270	240	210	410	200	260 ± 87
	Sum	270	290	220	530	280	320 ± 120
Diethylphthalate	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	52	ND	ND	41	29	25 ± 24
	Sum	52	ND	ND	41	29	25 ± 24
Phenanthrene	Probe + filter	230	2,700	1,400	3,400	2,400	2,000 ± 1,200
	Resin	2,900	1,800	1,800	2,200	1,400	2,000 ± 570
	Sum	3,100	4,500	3,100	5,600	3,700	4,000 ± 1,100
Di-n-butylphthalate	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	240	200	88	140	380	210 ± 110
	Sum	240	200	88	140	380	210 ± 110
Fluoranthene	Probe + filter	150	2,400	1,100	2,100	2,100	1,600 ± 940
	Resin	2,000	930	880	850	370	1,000 ± 610
	Sum	2,200	3,300	1,900	2,900	2,500	2,600 ± 560

(continued)

TABLE 44 (concluded)

Compound	Train component	Emission rate (mg/hr)					
		Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Pyrene	Probe + filter	190	3,700	1,100	2,600	2,200	2,000 ± 1,400
	Resin	2,300	1,100	960	890	360	1,100 ± 730
	Sum	2,500	4,800	2,100	3,500	2,600	3,100 ± 1,100
Butylbenzylphthalate	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	15	ND	21	ND	ND	7.3 ± 10
	Sum	15	ND	21	ND	ND	7.3 ± 10
Chrysene	Probe + filter	ND	200	120	310	460	220 ± 180
	Resin	160	41	59	62	23	69 ± 53
	Sum	160	240	170	380	480	290 ± 140
Bis[2-ethylhexyl]phthalate	Probe + filter	23	700	14	8.3	27	150 ± 300
	Resin	340	ND	7.7	66	120	110 ± 140
	Sum	370	700	22	74	140	260 ± 280
Benzofluoranthene	Probe + filter	35	300	150	440	500	280 ± 190
	Resin	2,000	39	61	83	22	440 ± 880
	Sum	2,100	340	210	520	520	730 ± 750
Benzo[a]pyrene	Probe + filter	29	220	120	280	340	200 ± 130
	Resin	130	25	31	41	9.4	46 ± 46
	Sum	160	250	150	320	350	240 ± 94
Dibenz[a,h]anthracene	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	ND	ND	ND	ND	2.6	0.52 ± 1.2
	Sum	ND	ND	ND	ND	2.6	0.52 ± 1.2
Benzo[g,h,i]perylene	Probe + filter	ND	ND	ND	ND	450	91 ± 200
	Resin	89	ND	33	ND	20	28 ± 37
	Sum	89	ND	33	ND	470	120 ± 200

a Mean ± standard deviation for the five tests.

b Not detected.

TABLE 45. EMISSION RATES FOR POLYCHLORINATED BIPHENYLS IN FLUE GAS

Homolog	Train component	Emission rate (mg/hr)					
		Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Monochlorobiphenyl	Probe + filter	ND[b]	0.42	0.34	1.0	0.12	0.38 ± 0.39
	Resin	ND	3.2	4.6	4.2	1.6	2.7 ± 1.9
	Sum	ND	3.7	4.9	5.2	1.7	3.1 ± 2.2
Dichlorobiphenyl	Probe + filter	1.5	3.0	0.58	8.2	0.69	2.8 ± 3.2
	Resin	ND	11	4.4	3.4	4.9	4.8 ± 4.1
	Sum	1.5	14	5.0	12	5.6	7.6 ± 5.3
Trichlorobiphenyl	Probe + filter	ND	1.6	3.8	1.2	0.81	1.5 ± 1.4
	Resin	ND	1.2	4.2	0.63	0.33	1.3 ± 1.7
	Sum	ND	2.8	8.1	1.8	1.1	2.8 ± 3.1
Tetrachlorobiphenyl	Probe + filter	ND	ND	0.67	0.41	ND	0.22 ± 0.31
	Resin	ND	0.51	0.84	ND	0.042	0.28 ± 0.38
	Sum	ND	0.51	1.5	0.41	0.042	0.49 ± 0.61
Pentachlorobiphenyl	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	1.2	0.017	0.28	0.43	ND	0.38 ± 0.47
	Sum	1.2	0.017	0.28	0.43	ND	0.37 ± 0.47
Hexachlorobiphenyl	Probe + filter	ND	0.26	ND	ND	ND	0.053 ± 0.12
	Resin	ND	0.011	0.17	0.59	ND	0.15 ± 0.25
	Sum	ND	0.27	0.17	0.59	ND	0.21 ± 0.24
Heptachlorobiphenyl	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	ND	ND	ND	0.017	ND	0.0033 ± 0.0075
	Sum	ND	ND	ND	0.017	ND	0.0033 ± 0.0075
Octachlorobiphenyl	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	ND	ND	ND	ND	ND	ND
	Sum	ND	ND	ND	ND	ND	ND

(continued)

TABLE 45 (concluded)

Homolog	Train component	Emission rate (mg/hr)					
		Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Nonachlorobiphenyl	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	ND	ND	ND	ND	ND	ND
	Sum	ND	ND	ND	ND	ND	ND
Decachlorobiphenyl	Probe + filter	ND	ND	ND	ND	ND	ND
	Resin	ND	ND	ND	ND	ND	ND
	Sum	ND	ND	ND	ND	ND	ND
Total chlorobiphenyl	Probe + filter	1.5	5.3	5.4	11	1.6	4.9 ± 3.8
	Resin	1.2	16	15	9.3	6.9	9.6 ± 6.1
	Sum	2.6	22	20	20	8.5	15 ± 8.5

a Mean ± standard deviation for the five tests.

b Not detected.

TABLE 46. EMISSION RATES FOR POLYCHLORINATED DIBENZO-p-DIOXINS IN FLUE GAS

Homolog	Train component	Emission rate (mg/hr)					
		Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Monochlorodibenzo-p-dioxin	Probe + filter	ND[b]	ND	0.023	0.20	ND	0.045 ± 0.089
	Resin	0.28	0.13	0.099	0.072	ND	0.12 ± 0.10
	Sum	0.28	0.13	0.12	0.27	ND	0.16 ± 0.12
Dichlorodibenzo-p-dioxin	Probe + filter	ND	ND	0.95	3.0	ND	0.78 ± 1.3
	Resin	0.55	ND	ND	ND	ND	0.11 ± 0.24
	Sum	0.55	ND	0.95	3.0	ND	0.89 ± 1.2
Trichlorodibenzo-p-dioxin	Probe + filter	ND	ND	1.7	3.1	0.38	1.0 ± 1.3
	Resin	ND	ND	ND	ND	ND	ND
	Sum	ND	ND	1.7	3.1	0.38	1.0 ± 1.3
Tetrachlorodibenzo-p-dioxin	Probe + filter	1.6	0.69	11	8.2	2.1	4.8 ± 4.7
	Resin	1.8	0.16	0.13	ND	ND	0.43 ± 0.79
	Sum	3.4	0.85	11	8.2	2.1	5.2 ± 4.4
Pentachlorodibenzo-p-dioxin	Probe + filter	14	5.6	71	34	9.0	27 ± 27
	Resin	8.7	ND	ND	ND	ND	1.7 ± 3.9
	Sum	22	5.6	71	34	9.0	28 ± 26
Hexachlorodibenzo-p-dioxin	Probe + filter	8.9	4.8	19	12	2.9	9.6 ± 6.5
	Resin	6.4	0.44	0.69	0.97	0.11	1.7 ± 2.6
	Sum	15	5.2	20	13	3.1	11 ± 7.0
Heptachlorodibenzo-p-dioxin	Probe + filter	3.9	1.8	5.2	3.4	0.76	3.0 ± 1.8
	Resin	1.8	0.087	0.13	0.38	0.042	0.48 ± 0.73
	Sum	5.6	1.9	5.3	3.7	0.80	3.5 ± 2.1
Octachlorodibenzo-p-dioxin	Probe + filter	1.3	0.42	0.87	0.73	0.27	0.72 ± 0.41
	Resin	0.63	0.013	0.031	0.15	0.016	0.17 ± 0.26
	Sum	1.9	0.43	0.90	0.88	0.28	0.88 ± 0.65
Total chlorodibenzo-p-dioxins	Probe + filter	29	13	110	65	15	47 ± 41
	Resin	20	0.84	1.1	1.6	0.17	4.8 ± 8.6
	Sum	49	14	110	67	16	51 ± 40

a Mean ± standard deviation for the five tests.
b Not detected.

TABLE 47. EMISSION RATES FOR POLYCHLORINATED DIBENZOFURANS IN FLUE GAS

Homolog	Train component	Emission rate (mg/hr)					
		Day 1	Day 2	Day 3	Day 4	Day 5	Mean[a]
Monochlorodibenzofuran	Probe + filter	1.5	0.56	1.5	3.1	2.4	1.8 ± 0.99
	Resin	6.5	7.6	6.0	6.2	3.4	5.9 ± 1.6
	Sum	8.0	8.2	7.4	9.4	5.8	7.8 ± 1.3
Dichlorodibenzofuran	Probe + filter	2.1	3.6	7.1	12	7.3	6.5 ± 4.0
	Resin	6.3	6.4	5.3	3.2	1.1	4.5 ± 2.3
	Sum	8.4	10	12	16	8.4	11 ± 3.1
Trichlorodibenzofuran	Probe + filter	12	14	45	67	29	33 ± 23
	Resin	25	9.4	7.6	8.0	1.5	10 ± 8.9
	Sum	37	23	52	75	31	44 ± 20
Tetrachlorodibenzofuran	Probe + filter	6.9	7.2	47	32	10	21 ± 18
	Resin	9.7	2.6	2.8	2.5	0.18	3.6 ± 3.6
	Sum	17	9.8	50	35	11	24 ± 17
Pentachlorodibenzofuran	Probe + filter	30	22	370	200	54	130 ± 150
	Resin	29	4.1	5.2	7.8	0.37	9.3 ± 11
	Sum	58	26	370	210	55	140 ± 150
Hexachlorodibenzofuran	Probe + filter	ND[b]	3.5	46	21	6.3	15 ± 19
	Resin	4.4	0.0030	0.11	0.55	0.095	1.0 ± 1.9
	Sum	4.4	3.5	46	21	6.4	16 ± 18
Heptachlorodibenzofuran	Probe + filter	2.9	2.0	9.3	4.9	1.5	4.1 ± 3.2
	Resin	1.6	0.13	0.17	0.32	0.068	0.46 ± 0.65
	Sum	4.5	2.1	9.5	5.3	1.6	4.6 ± 3.2
Octachlorodibenzofuran	Probe + filter	0.17	0.18	0.60	0.41	0.17	0.31 ± 0.19
	Resin	ND	ND	ND	ND	ND	ND
	Sum	0.17	0.18	0.60	0.41	0.17	0.31
Total chlorodibenzofurans	Probe + filter	55	53	520	340	110	
	Resin	83	30	27	29	6.7	3
	Sum	140	83	550	370	120	25 ± 200

a Mean ± standard deviation for the five tests.

b Not detected.

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